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Contractor Report - ARCCD-CR-86002

MAGNESIUM FLUORIDE REDUCTION VESSEL LINERS

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Prepared for

Department of the Army  
U.S. Army Research and Development Command  
Dover, New Jersey 07801

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## INTRODUCTION

### OBJECTIVE OF THE PROGRAM

The objective of the efforts described in this report is to replace the graphite liner currently being used in the production of depleted uranium derby. Utilization of magnesium fluoride ( $MgF_2$ ) as a reduction vessel liner has the potential to decrease carbon contamination and thereby reduce DU derby rejects due to chemistry. Additionally there would be the elimination of the cost of the graphite crucible liner and the associated disposal costs by replacement with the by-product of the reduction reaction, which is magnesium fluoride. The process would ultimately result in reduced manufacturing costs for derby metal and higher yield of finished penetrators.

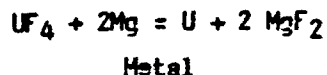
### SCOPE OF THE MANUFACTURING METHOD TECHNOLOGY PROGRAM

The scope was established to replace the dolomite and graphite presently used to line reduction vessels with magnesium fluoride for the reduction of uranium tetrafluoride ( $UF_4$ ) to uranium metal. This was to be accomplished in such a manner as to produce uranium metal derbies which would be accommodated into the present Nuclear Metals-Carolina Metals penetrator production process with minimal changes in equipment and procedures.

## BACKGROUND

A procedure is presently in use at the Feed Materials Production Center (Fernald, Ohio) whereby the magnesium fluoride produced in the reduction reaction is utilized as liner material. Therefore, this program is designed to address the factors which will lead to modifications of the NMI reduction procedure resulting in the utilization of the by-product material for reduction vessel liners and the associated benefits derived by this change.

The reduction process consists of blending properly sized  $UF_4$  and magnesium, placing the blend in a closed, lined, steel vessel, and setting off an exothermic reaction by heating the vessel in a furnace to approximately  $700^\circ C$  ( $1300^\circ$ ).



The procedure in use at the Feed Materials Production Center uses a long tapered retort and mandrel. The charge when fired produces a derby in the range of 300 lbs.

The general procedure in producing the 300 lb derby is as follows:

- A - Grind, screen for size, and blend the required percentages of  $MgF_2$  prior to the formation of the liner.
- B - Insert the mandrel into the retort; fill and jolt the assembled unit to compact the liner material.
- C - Remove the mandrel and charge with the required blend of  $UF_4$  and Mg.
- D - Seal the vessel and place in a furnace to initiate the reaction. Firing time required is approximately four hours.
- E - Cool; remove cover plate and invert and jolt to break out derby and  $MgF_2$  by-product.

In the work to be carried out for this program at Nuclear Metals, Inc. a different set of problems are required to be addressed. The configuration of the retort vessel is entirely different than those used at FMPC. Therefore the formation of a  $MgF_2$  liner in this different design must be examined and physically tried in order to determine the packing characteristics. While the blend of the  $UF_4$  and Mg is the same, the volume of the charge is considerably greater. The weight of the derby produced with this vessel is in the order of 1400 lbs. The firing time to induce the reaction for such a charge is between seven and eight hours.

From the standpoint of the physical size of the derby produced, the economics favors the development for a method using a larger charge per reaction. It is this very point that required the examination of those conditions permitting the processing of large reductions in which cost savings would be reflected.

Figure 1A shows the reduction vessel presently used at Carolina Metals, Inc. (CMI), a wholly owned subsidiary of Nuclear Metals, Inc. The graphite liner in which the reduction reaction takes place is illustrated in Figure 1B. Dolomite is placed on the bottom of the steel vessel and centered with the space between the outer graphite and the inner steel wall being filled with dolomite using electro-mechanical vibrators.

Once the graphite liner is in place, it is filled with the  $UF_4$  - Mg blend. To complete the assembly of the charge, a graphite plate covers the blend and a dolomite cap is formed over the plate. The retort is then sealed with a steel cover bolted in place. Once assembled, the unit is placed into the furnace and heated to a temperature sufficient to initiate the exothermic reaction. Once the reduction has taken place, the vessel is allowed to cool prior to the removal of the steel cover plate. A retaining ring is placed over the dolomite and graphite liner after which the vessel is inverted and the reacted charge consisting of the derby metal and the resulting magnesium fluoride by-product is removed by jolting.

All of the  $MgF_2$  as well as the dolomite from the cap and the graphite is discarded.

#### METHODOLOGY AND EXPERIMENTAL WORK

##### MAGNESIUM FLUORIDE POWDER PREPARATION

The packing characteristics of powdered material is affected by both the particle sizes and the particle shape.



In the early stages of this program, some material from the Feed Materials Production Center (NLO) was used for comparison testing. Under their program, the magnesium fluoride from the breakout stations is rough screened and ball milled for various lengths of time to achieve the desired degree of fineness. The actual screening of the fractions is accomplished by means of an air classifier.

Reduction of the magnesium fluoride to a powder form at Carolina Metals is accomplished through the use of a hammer mill. The material so reduced is then separated into the various sizes through a screening operation.

In order to compare the particle shapes developed by ball milling against the hammer-mill, samples of both were prepared for examination by means of a scanning electron microscope. Considering the range size with which we are concerned, we elected 50X, 200X, and 2000X as the magnifications which would give the best basis for comparison.

The examination of the pictures in the two sets of figures nos. 2, 3, and 4 indicates a somewhat more rounded particle is produced in the ball milling operation. The shape produced by the hammer mill shows a generally sharper configuraion. However, in actual practice it is not possible to ascertain any real difference in the resultant formation of liners that one could attribute to this factor.

#### TEST PROCEDURES

In order to form a liner of magnesium fluoride, it is necessary not only to reduce the material to a powder form, but also to classify the powder according to size fractions.

A typical screen analysis of material from the Feed Material Production Center (NLO) is as follows:

Screen	-325	+325	+200	+100	+60	+40
Wt. %	38	15	23	15	8	1

Through experiments conducted at the FMPC Laboratory, it was determined that when material was jolted, the best characteristics were obtained when the mix consisted of the following size fractions:

Screen	-325	+325 -200	+200 -60
Wt. %	45	35	20

In comparison, of the samples prepared by Carolina Metals, the best results were obtained with the following blend: (Reference Table 1)

Screen	-325	+325 -200	+200 -60
Wt. %	65	25	10

At Carolina Metals, the fluoride is reduced to power by means of a Mikro #2 hammer mill (Figure 5). The material is then separated into the different size fractions by means of a Sweco Classifier (Figure 6) into the same ranges as those produced at the Feed Materials Production Center (NLO). Specific blends of the particle sizes were prepared to determine the effect of compacted surface hardness when subjected to various applied pressures.

To accomplish this, a small slightly tapered cylinder was prepared into which the powder blends were packed. The procedure consisted of filling the die and tapping the side until the powder appeared not to settle any further. At this time, a surface hardness reading was made using a Dietert-Detroit Model 473 "Green Hardness" tester. The hardness values are relative with no relationship to any particular scale. The sole purpose of this type of data is to permit a comparison of the surface condition developed as a function of the packing technique. This is precisely the same method employed in foundries to determine surface hardness on sand molds.

Each succeeding test was done in the same manner, filling the die cavity with the powder blend, tapping the side to first settle the powder and then applying compression through a hardened plug at one end of the cylinder cavity. While the numbers are relative, Table I indicates a difference in surface hardness. This difference is attributed to the composition of the blends tested.

Figures 7 and 8 show the laboratory press and the resulting core upon its removal from the die. All measurements of surface hardness were taken while the powder was contained in the packing die. Unsupported, the core has virtually no green strength.

The complete series of surface hardness values are listed in Table 1. This data generated by the application of direct pressure was only for the purpose of establishing values relative to the combinations of the particle sizes. Actual packing in the formation of a liner would be accomplished by the use of a jolting or vibration mechanism.

The magnesium fluoride liners are formed without binder material. The green strength of the powder is a function of the particle size distribution and the means employed to effect compaction.

#### REDUCTION MANDREL/RETORT DESIGN

In order to conform to the physical size of the DU metal derby produced at CMI as illustrated in Figure 18, the initial liner mandrel was designed to the same dimensions as the inside of the graphite liner. This would therefore result in a  $MgF_2$  liner whose shape and thickness would be equal to that of the graphite plus the dolomite (Figure 9).

Based on the compacting trials as detailed in Table 1, the indication was that a blend consisting of the following  $MgF_2$  mesh sizes would result in the most dense liner when processed.

Screen	-325	+325 -200	+200 -65
Wt. %	65	25	10

The blending of this combination was accomplished by weighing the relative size proportions into a 55 gallon drum in small lots and then using a rotating head, fork lift truck for mixing. The direction of rotation was alternated some fifty times to insure as complete and uniform a mix as possible.

Since design work required that the facilities presently in use by CMI be utilized with minimum disruption, a hopper was designed to permit continuous filling of the retort with the  $MgF_2$  powder as a jolting action was imparted to effect compaction. Figure 11 pictures the unit being positioned within the confines of the vented room for filling and assembly.

The hopper is bolted to the steel retort and the unit is then positioned on the jolting table. The table is activated by air pressure. The limiting factor of this operation was the air tank reserve which resulted in some interruption of the packing cycle when the pressure dropped below the minimum required to effect the table action.

Following the particular prescribed jolting period, the hopper unit is removed and any excess  $MgF_2$  material is vacuumed from the distribution cone. The  $MgF_2$  thus consolidated between the mandrel and the retort shell comprises the liner as shown in Figure 12.

While the direct pressure compacting tests (Table I) shows that the mixtures containing a high percentage of the finer fraction results in a higher relative number of surface hardness, it does not translate directly into through hardness of the packing. It was decided to prepare a second blend which was to be weighted towards the courser fractions. The size distribution of the first blend, CMI mix A, and the second blend, CMI mix B, are shown below:

<u>CMI Mix A</u>		<u>CMI Mix B</u>	
65 wt. %	-325 mesh	40 wt. %	-325 mesh
25 wt. %	-200 mesh	35 wt. %	-200 mesh
10 wt. %	- 65 mesh	25 wt. %	- 65 mesh

Since the compaction of the  $MgF_2$  was to be accomplished by a jolting action rather than direct pressure, the compaction of the powder would differ. As the program continued and the method of compacting the liners was adjusted from the jolting or simulated slow vibration to a higher frequency vibration, indications were towards a more uniform density of the liner.

Therefore, on the actual reduction test liners as reported in Table 3, while both CMI mix A and mix B were used in the various combinations of retorts and mandrels, the best results were obtained using mix B in the modified taper assembly.

## DISCUSSION OF EXPERIMENTS

Table 2 is a compilation of all of the full size and reduced size packing tests carried out in this program. The tests have been numbered 1 through 34, and these numbers will be used throughout the remainder of this report to refer to the specific experiments carried out.

A series of trials using the normal CMI retort vessel, Figure 1A and a mandrel conforming to the inner dimensions of the graphite line, Figure 1B, were conducted. (Test Nos. 1, 2, 3, 4, 5, and 32.) For purposes of clarity, these forms will be referred to as standard in this text.

The first of the series were conducted using the jolting table for compaction. The frequency and packing times were varied as shown in Table 2. Considering the physical size and weight of the retort, mandrel, and the hopper with its charge of  $MgF_2$ , the jolting action was rather severe. Upon the partial withdrawal of the mandrel, there was an almost immediate collapse of the sidewall of the liner. (Figures 13 A and 13B)

Variations in frequency and packing times did not result in any improvement. In those instances where some of the sidewall integrity was maintained, surface hardness readings were within a range that should have produced a better result. Closer examination after vacuuming out collapsed material revealed that the initiation of the rupture was taking place at the transition angle between the upper and lower sections of the liner. The conclusion, therefore, was that the heavy jolting action, while seeming to effectively pack the vertical portion of the wall, was not uniformly translated throughout the liner leaving a soft section. Consequently, this soft section was unable to sustain the weight of the upper portion, resulting in the collapse.

Subsequent tests were carried out using heavy duty vibrators at 60 degree intervals about both the upper and lower sections of the retort. While there was some improvement in the packing characteristics, the transition angle remained the major obstacle to a uniform packing density. The end result was still a soft section which was unable to support any load. Reference Table 2, test nos. 1, 2, 3, 4, 5, and 32.

The physical preparation and work required to form a full size liner in this manner is considerable. In the interest of developing a modified configuration which would permit a more uniform translation of packing pressure, it was decided to fabricate 1/4 scale retorts and mandrels.

#### STRAIGHT TAPER REDUCED SCALE MANDREL

Three different 1/4 scale configurations were fabricated. The first was a long slender taper which was to serve as a guide to packing hardness resulting first from a jolting action and then vibration. The simple holding mechanism and form is shown in Figure 14. The unit consists of an outer casing within which the mandrel is centrally positioned with appropriate hold downs. The  $MgF_2$  blend was loaded to the top of the cylinder and vibrated down. As the material settled, more of the blend was added. This resulted in a solid liner which did not collapse. (Tests 9, 11, and 12.) There was no indication of a layered seam as the result of this method of filling when the mandrel was withdrawn.

Figures 14 and 15 show the withdrawal of the mandrel and the resultant liner. (Figure 16) The liner was deliberately mishandled and bumped to establish the quality of the packing. It survived all the testing which was considered to be well in excess of what might be encountered in normal handling procedures. (Reference Table 2, Tests 9, 11 and 12.)

## REDUCED SCALE STANDARD CMI GEOMETRY

A 1/4 scale retort and mandrel were prepared of the standard CMI vessel, Figures 1A and 1B. This was done to permit comparison of a stepped design with a tapered liner. Compacting by jolting alone, vibration alone, and in combination were all tried with the same negative end results as in the full scale trials. The inability of the  $MgF_2$  blend to flow and compact to a firm enough degree at the sharp transition angle to sustain the weight resulted in sidewall collapse upon even partial withdrawal of the mandrel. (Figures 17, 18 and 19.)

Repeated attempts were made with the 1/4 scale standard retort and mandrel using all the possible combinations to compact and overcome the transition angle problem but to no avail. Results are listed in Table 2, tests nos. 6, 7, 8, 10, 13, 14, 15, 16, 17, 18, 19, 20 and 28.

## 1/4 SCALE MODIFIED TAPER MANDREL

In appreciating the problem of the sharp transition angle, it was decided to make a 1/4 scale form of such a design as to take into account the required derby cavity and the reaction furnace limitations. The objective of this mod-1 would be to permit a free uniform flow path to effect a dense pack. The resulting simple form is illustrated in Figure 20.

To further understand the flow and compaction problem, the 1/4 scale CMI stepped retort was mated with the new taper mandrel. Table 2, test nos. 21, 22, 23, 24 and 25 all show good packing characteristics even with the extra thickness of material at the transition of the retort. Tests 26, 27 and 29 made with the combination of the taper retort and taper mandrel all showed excellent results. Figures 21 and 22 show the withdrawal and relative stability of one of the first tests. Based on this data, a full size retort and mandrel was designed and fabricated. (Figures 23 and 24.)

As would be necessary with any mandrel used in the formation of a liner, a vent tube "A" as shown in Figure 24 must be provided to relieve the vacuum to permit withdrawal. Paper tape is used to seal the opening during the formation of the liner. The insertion of a simple rod is all that is required to break the paper tape seal.

#### FULL SIZE EXPERIMENT

Figure 25 pictures the full size retort and mandrel. The mandrel flange mates with the retort and thereby serves to center the unit as well as establishing a proper distance from the bottom, allowing the  $MgF_2$  powder to form the liner bottom of the desired thickness.

Based on the trial program, the best packing resulted from the use of vibrators as compared to the jolting action. Heavy duty vibrators were fastened to the stiffening webs of the retort. The vessel was filled manually as the  $MgF_2$  powder compacted and the level dropped.

Table 2, test nos. 30, 31, and 33, all resulted in good liners being formed. Purely as a trial to bear out results of the 1/4 scale combination of the standard CMI retort and taper mandrel, the full size units were mated and packed. Test number 34 of Table 2 again shows the beneficial effect of a free flow.

#### REDUCTION VESSEL PACKING

Following the mating of the retort and mandrel, the formation of the liner is completed in accordance with the packing times as listed in Table 3 titled "Reduction Test Liners". The series of pictures present an outline of the steps involved in the physical procedure up through the knockout of the derby following the reduction reaction.



The removal of the mandrel after the formation of the liner requires a straight pull to prevent contact with the sidewall. Centering rods are positioned to assist in alignment during mandrel withdrawal. Figure 26 shows the chain fall attachment to the distribution cone. In Figure 27, the firm, well formed liner is readily visible with the partial withdrawal of the mandrel.

The vessel with its formed liner is then transported by fork truck to the cabinet beneath the blending station as shown in Figure 28. It is at this point that the  $UF_4 + Mg$  is introduced to the "V" mixer shown on the upper level.

After the prescribed mixing time, a snorkel fill tube is attached to the discharge port of the mixer. The blend is introduced to the bottom of the liner first so as to not impact on the liner wall. The snorkel is gradually withdrawn as filling continues. The blend material now becomes the support for the  $MgF_2$  liner and liner integrity is maintained for the balance of the operation.

The initial attachment of the snorkel tube is pictured in Figure 29 and the positioning of the retort prior to filling is shown in Figure 30.

Once the filling of the  $UF_4 + Mg$  blend has been completed, the vessel is moved to a capping station where it is leveled and tapped firm. For safety reasons and because the procedure is still experimental in nature, a graphite cap was placed over the charge prior to sealing the vessel with a steel cover. In a production mode, the cover material would be  $MgF_2$ , thereby eliminating all graphite from the vessel.

The prepared vessel is transported from the capping station to the furnace liner where it is lowered into the firing pit. Figures 32, 33 and 34 show the handling procedures.

The capped vessel can now be handled in the same manner as the regular CMI retorts for processing. Figure 31 pictures the furnace line at Carolina Metals. The furnaces are below floor level and are programmed to first pre-

heat and at the appropriate time, to increase the power input to the point of ignition of the blend with the resultant effect of producing DU derby metal and the by product  $MgF_2$ .

Following the reaction, the vessel is removed from the furnace and after an initial cooling period, is processed through a cooling chamber. The retorts are designed in such a manner as to have handling dogs on each side as shown in Figure 35.

The final phase of the operation is the removal of the solid derby metal from the retort. The jolter table previously referred to in the liner processing operation is the knockout station. As shown in Figure 36, the vessel is inverted and placed on the table. It is not necessary to bolt the vessel to the table as the weight will hold it in position. It is usually only necessary to jolt the unit a few times before the derby breaks loose taking the  $MgF_2$  with it into the catch basin below. The  $MgF_2$  is carried off on a belt conveyor to a jaw crusher and automatically dumped into 55 gallon drums for processing or disposal.

#### REDUCTION PROVEOUT TESTS

A series of test liners were prepared in accordance with the procedure as outlined in Figure 37,  $MgF_2$  Liner Derby Flow Sheet. Up to this point the main concern was the fabrication of an acceptable liner; now it was necessary to follow through with the actual reduction reaction. The difference in the configuration between the standard CMI reduction vessel (Figure 1A) and the modified taper retort (Figure 23) suggests that some modification to the heating cycle might be necessary. Heating times are well established for the CMI standard retort and its graphite liner with dolomite insulation as shown in Figure A. In the case of the liners produced and used as part of the actual proveout tests, we encounter a condition whereby some soak time allowance is necessary to accommodate a variation in  $MgF_2$  wall thickness and its insulating property.

The modified taper mandrel when used in conjunction with the standard CMI retort results in a heavy  $MgF_2$  section at the transition angle as illustrated in Figure B.

The combination of the taper mandrel with the taper retort as shown in Figure C allows for a wall thickness equal to the graphite liner plus the dolomite packing at the lower section and results in a thinner section at the upper reaches of the assembly.

The "X" series, as shown in Table 3, were processed to permit evaluation of soak and reduction reaction times. A standard heat cycle will vary between 7 and 8 hours to the point of ignition. Because of the heavier side wall on the modified retort, the decision was made to extend the pre-heat time. The modified taper retort was heated to 700°F for five hours, after which the temperature was increased to 1300°F to effect ignition. By virtue of this adjustment, the total time extended to between 11 and 12 hours. Under actual production conditions, this time would be reduced through the use of a thinner wall,  $MgF_2$  liner.

A total of ten reductions were carried out to demonstrate capability of processing. Photographs of the proveout liners are shown in Figure 38. From the latter portion of the proveout process, three derbies were selected to be processed in accordance with the Process Control Document No. 833-051 as outlined in Figure 39.

As noted in Table 3, liners x-2 and x-4 were processed using the CMI outer vessel and the tapered mandrel. This assembly results in a much thicker wall at the transition angle. Since no time adjustments were made in compacting the liners, it was expected that this area would not be as firm.

Generally the overall appearance of the formed liners was good. There were a few small breaks as noted at the transition angle. Surface hardness readings taken were in the range of those achieved in the test program.

When the reductions were made, some of the derbies had very poor, rough, irregular surfaces. We believe this condition was due to a breakaway of the liner wall which occurred during the violent reaction of the reduction process.

The use of better packing equipment such as that used at FMPC would result in a harder liner giving derby surfaces equivalent to those produced in the present graphite liners.

Packing times will require further investigation to determine the depth of the hardened liner wall necessary to eliminate this problem.

#### CHEMISTRY

In the normal processing of DU derby in a graphite lined vessel, the metal is subject to contamination from three different sources. Iron is found to be present in both the  $UF_4$  and the Mg. Iron analysis on Mg as purchased can range from 20 ppm to 50 ppm.  $UF_4$  is not analyzed for iron except under special circumstances. The carbon contamination comes from the graphite liner.

In reducing the  $MgF_2$  chunks to powder by use of the hammer mill we introduced another source of iron contamination. The hammers themselves are made of a wear resistant hardened steel, however, due to the abrasive nature of the  $MgF_2$ , the wear on the surfaces was still quite pronounced.

The  $MgF_2$  produced in the reaction, when broken out after the reduction, is of two distinct colors. That portion which was in direct contact will vary from black to light grey. The  $MgF_2$  from the center section varies from light grey to a crystalline white. The darker material is higher in carbon.

The liners for the first reduction were prepared without regard to the color of the material reduced to powder form. Analysis of the derby produced in these liners showed medium high iron content and carbon results higher than expected.

The later  $MgF_2$  liners were prepared by selecting only the white portions from a number of reductions and reducing this material to powder form. Derby produced under these conditions showed some reduction in iron but primarily a sharp drop in carbon values. If graphite is eliminated from the reduction process and replaced by  $MgF_2$ , no source of carbon would be present and the  $MgF_2$  used for the liners would not have to be chosen selectively.

Reduction of the  $MgF_2$  to the powder form by using a ball mill would result in the elimination of the iron contamination associated with the hammer mill. Such a mill would require a ceramic liner and the use of ceramic balls as the grinding medium.

On the three prove out melts, x-7, x-8, and x-10, chemistry was performed on the extruded rods in accordance with MIL-C-63422 as reported in Table 4 at the positions indicated by the sample number on each of the three lots.

The analysis of the elements listed were done using the following methods:

- A. Ti was determined colorimetrically
- B. C by combustion with infra-red detection of  $CO_2$
- C. Fe, Ni, Si and Cu by atomic absorption after separation from uranium
- D. Mg determined directly by atomic absorption
- E. F determined by direct potentiometry using a selective ion electrode

#### ULTRASONIC INSPECTION

In accordance with MIL-C-63422, paragraph 4.5.7, we conducted a 100% inspection of the blanks generated from the three billets extruded from each of the prove out melts, x-7, x-8, and x-10. The tests were run manually with each rod having its own printout.

The ultrasonic tester is a four channel system whereby the left and right transducers are set at a pre-determined angle. The pulse echo transducer is on the centerline and the fourth transducer is offset .273" from the centerline.

Lot x-7 consisted of 31 pre-machined blanks from which there were no rejects. Lot x-8 contained 33 blanks from which one piece was rejected for a surface defect. Lot x-10 resulted in 32 blanks being prepared and there were no rejects.

Comparison of this data with normal production melts places the results in an above average category. Ultrasonic reject rates for M833 production is in the range of 2 to 3% while the reject rate for material produced under this contract was about 1%. However, the size of the sampling is not sufficiently large to permit a definite conclusion.

#### METALLOGRAPHY

Metallographic examination of the extruded material was carried out on the front and rear sections of one rod from each of the three lots.

The following series of photomicrographs are the edge, mid-radius, and center sections at magnifications of 50x, 100x, and 250x. In each case, what is revealed is a basic acicular alpha prone structure with no precipitates in the grain boundaries and good through transformation. This is a typically normal structure for this alloy. Inclusions are small and consist of carbides and oxides. This is quite normal for the material.

The overall quality of the material illustrated is considered to be equal to or generally better than that normally seen in standard production material. Specifically, the inclusions were smaller and fewer in number than usually encountered in routine examinations.

## **MgF<sub>2</sub> LINER ECONOMIC ANALYSIS**

### **PURPOSE**

The purpose of this analysis is to determine the effect on penetrator cost of performing reductions of UF<sub>4</sub> to depleted uranium (DU) derby using MgF<sub>2</sub> liners instead of graphite liners. The technical feasibility of this effort was demonstrated under the Manufacturing Methods and Technology (MM&T) Contract DAAK10-83-C-0276.

### **OPERATION DESCRIPTION**

NMI's current method of producing DU derbies was utilized in this analysis. A detailed description of this process is found in NMI's Process Control Document for M833 cores NMI 83-0063 PCD, Revision B dated 5-18-84. The process is briefly summarized below:

#### **1. Preparation of the Reduction Vessel**

- New retorts and graphite liners are coated then dried.
- Graphite liner is placed inside the metal retort and the space between the retort and liner is filled with dolomite or ground MgF<sub>2</sub> and then packed.
- Previously used retorts are burned out, then scraped, cleaned, and re-coated.

#### **2. Mixing and Loading of the Reduction Vessel**

- The prepared vessel with liner is positioned under the blender and a mixture of UF<sub>4</sub> and magnesium granules is charged into the retort.
- The charged vessel is then packed, capped with a graphite disc of dolomite or MgF<sub>2</sub>, then a cover is bolted to the vessel.

OPERATION:  $MgF_2$  Liners

DESCRIPTION:

$MgF_2$  Liner Preparation:

- o Grind/Screen  $MgF_2$  using Ball Mill and Classifying Screens with 62, 200, 325 mesh.
- o  $MgF_2$  Liner Charging Station for charging ground  $MgF_2$  into metal retort with mandrel using vibrators to pack and form the liner.
- o  $UF_4$  and Mg Blending Station with telescope charging chute and 2-ton overhead hoist for removing mandrel prior to charging retort with liner.

OPERATION PARAMETERS:	EQUIPMENT REQUIREMENTS		
	Required	In-house	Add'l
1.20" $\phi$ : 243 liners x 2 hr/liner stations    350 hr = 1.39	2	0	2
1.15" $\phi$ : 225 liners x 2 hr/liner stations    350 hr = 1.28	2	0	2
1.10" $\phi$ : 205 liners x 2 hr/liner stations    350 hr = 1.17	2	0	2

ASSUMPTIONS/NOTES:

Liner Preparation Yield: 90%

Operation Efficiency: 70% (3-8-5 shift) = 350 hrs/mo

Derby Yield: 94%

Composition of Liner Preparation Station:

- Ball Mill Grinder and Screening Classifier for 30,000 # $MgF_2$  per day capacity
- $MgF_2$  Liner Preparation:
  - Hopper, charging chute, ventilation
  - Vibrators (3) and vibrating table for each station
  - Mandrel (8) @ \$9,400 ea.
  - Metal Retort (24) @ \$2,900 ea.
- $UF_4$  and Mg Blender w/charging chute and 2 ton hoist

Est. Cost  
\$30,000

10,000

7,500

75,200

69,600

50,000



### 3. Reduction of $\text{UF}_4$

- The loaded vessel is then placed in the reduction furnace and brought up to temperature during which time a reaction occurs.
- The vessel is removed to a cooling station, cooled, vessel is opened, vessel inverted and placed on a jolter, and the derby is removed.
- The derby is then weighed, stamped, analyzed for chemistry, pickled, rinsed in water, re-weighed, packaged, and shipped.

The  $\text{MgF}_2$  liner process only differs from the graphite liner process in vessel preparation and mixing and loading of the vessel. These differences are briefly described below:

#### 1. Liner Preparation

- $\text{MgF}_2$  is ground on a ball mill and classified by size on various screens.
- A mandrel is suspended in a reduction vessel and ground  $\text{MgF}_2$  is charged into the retort to form the liner. The compaction of the  $\text{MgF}_2$  is accomplished by means of a jolter/vibrator.

#### 2. Mixing and Loading of the Reduction Vessel

- The mandrel is removed from the retort and the liner is inspected for cracks.
- The mixture of  $\text{UF}_4$  and magnesium granules is charged into the vessel. A telescopic chute is used to charge the vessel to minimize damage to the integrity of the  $\text{MgF}_2$  liner.
- The vessel is then packed, capped with additional ground  $\text{MgF}_2$ , and then a cover is bolted to the vessel.
- The vessel then would await the initiation of the standard reduction process.

## ASSUMPTIONS

The economic analysis was performed using the following assumptions:

1. The process of preparing the  $MgF_2$  liner has been refined and de-bugged to the point that no further experimentation is needed to meet the needs of production.
2. Production equipment to grind and classify the  $MgF_2$ , prepare the  $MgF_2$  liner, and charge the vessel with  $UF_4$  and  $Mg$  has been procured, installed, and accepted. The type and mix of equipment utilized would be similar to that already in operation at FMPC. Briefly, the equipment would be comprised of the following:
  - An integrated  $MgF_2$  grinding station consisting of a ball mill, conveyor system, classifying screens, and storage bins.
  - A liner preparation station adjacent to the  $UF_4$  and  $Mg$  blending station consisting of a ground  $MgF_2$  overhead hopper, housings for the metal retort, overhead crane for the mandrel, and a jolter/vibrator platform.
  - A  $UF_4$  and  $Mg$  blending station integrated with the liner preparation station to remove the mandrel from the liner by means of an overhead crane, and then charging  $UF_4$  and  $Mg$  into the reduction vessel.
3. Utilization of  $MgF_2$  liners will produce a slight increase in derby yield of 2%.
4. Utilization of  $MgF_2$  liners will produce a slight increase in rod chemistry carbon (C) yield of 2%.
5. Reduction firing times of vessels with  $MgF_2$  liners will be the same as the current process which uses graphite liners.
6.  $MgF_2$  cap in lieu of a graphite cap would be used for each reduction.
7. Once a steady-state amount of ground  $MgF_2$  is available, not all  $MgF_2$  generated in the reduction process will be utilized. Some  $MgF_2$  will be buried. Approximately 2,000 lbs. of ground  $MgF_2$  will be needed per reduction. Each reduction generates approximately 800 lbs. of

new  $MgF_2$ . Assuming that 10% of the liner material cannot be reused and must be buried, then only 200 lbs. of new  $MgF_2$  is needed to replenish the supply of available ground  $MgF_2$ . Therefore, once steady-state is achieved, approximately 800 lbs. of  $MgF_2$  would be buried per reduction (200 lbs. old  $MgF_2$  plus 600 lbs. new  $MgF_2$ ). It is important to realize that this is the same amount of  $MgF_2$  as produced with a graphite liner.

## COST ANALYSIS

### Major Cost Elements

1. The parameters of cost affected by the proposed process are listed below:

#### a. Estimated Labor Hours Per Derby

<u>Operation</u>	<u>Graphite Liner</u>	<u><math>MgF_2</math> Liner</u>
Liner Preparation	3.5 hr.	3.3 hr.
Weigh Charge to Blender	0.5 hr.	0.5 hr.
Charge Vessel	0.3 hr.	0.7 hr.
Compact/Cap Vessel	0.1 hr.	0.1 hr.
Firing Time	<u>7.5 hr.</u>	<u>7.5 hr.</u>
	11.9 hr.	12.1 hr.

### Material

#### 1. Current Process:

- Graphite liner (assume 14 reductions per liner).
- Coating for liner and retort.
- Graphite disc for cap.

#### 2. $MgF_2$ Process

- No material required.

### 3. Waste Disposal

#### a. Graphite Liner Method

- Graphite liner disposal 2-55 gallon drums.
- 800 lbs.  $MgF_2$  per reduction or .88-55 gallon drums per reduction.

#### b. $MgF_2$ Liner Method

- 800 lbs.  $MgF_2$  per reduction or .88-55 gallon drums per reduction.

### ECONOMIC ANALYSIS

1. Assuming it takes 53 derbies to finally yield 5,000 finished cores, it is expected that with refinement of the  $MgF_2$  liner process, the derby chemistry yield should increase by 2% due to fewer iron and carbon rejects. Correspondingly, a redesigned retort and mandrel should increase derby yield (weight) by 2% from an average weight of 1354 lbs/derby to 1382 lbs/derby based upon  $MgF_2$  being a ceramic considered an insulator and carbon (graphite) being considered a conductor.

#### a. Liner Costs

	<u>Graphite Liner</u>	<u><math>MgF_2</math> Liner</u>
Labor:	\$ 77.03/derby	\$ 77.61/derby
Material:	\$291.00/derby	\$ 0 /derby
(graphite liner)		
Waste:	\$ 23.32/derby	\$ 0 /derby
(graphite liner)		
	• 56.4 derbies	• 54.6 derbies
	<u>to yield 53</u>	<u>to yield 53</u>
Cost Difference:	\$22,072	\$4,237

b. Calculation of Cost " " per lb. of derby:

Graphite Liner Method

MgF<sub>2</sub> Liner Method

(1) 53 derbies  
@ 1354 lb/derby =  
71,762 lb.

53 derbies  
@ 1382 lb/derby =  
73,246 lbs.

(2) \$22,072/71,762 lbs.  
\$ 0.3076/lb.

\$4,237/73,246 lbs.  
\$0.0579/lb.

c. Cost Savings per pound of derby:

$$\$0.3076 - \$0.0579 = \$0.2497/\text{lb. of derby}$$

\$0.2497 represents the cost savings per pound of derby using the MgF<sub>2</sub> liner method.

A \$0.25 cost savings per pound of derby is a 14.5% reduction in the current cost per pound of derby. Assuming that Carolina Metals produces over 3,000,000/lbs. of derby per year at \$0.25 per pound, cost reduction equates to a potential \$750,000 annual cost savings in derby production alone.

2. Cost Per Penetrator

The cost per penetrator was calculated using the following criteria:

- a. Current M833 yields by operation except for a 2% increase in rod chemistry due to fewer carbon rejects.
- b. Standard M833 hours.
- c. Standard M833 Bill of Materials except for a \$.25 per pound reduction in the cost of derby.
- d. 53 derbies needed to produce 5,000 M833 penetrators.

The above noted criteria was loaded into NMI's computer program for calculating the cost per penetrator. A cost savings of \$6.87 was realized. The major cost elements which were affected are as follows:

1. Material cost for processing one less melt lot due to a 2% increase in rod chemistry yield and reduced derby cost of \$.25 per pound.
2. Labor cost for processing one less melt lot through rod chemistry. An increase in yield at rod chemistry would require one less starting derby to achieve the required 5,000 M833 penetrators.

#### FACILITIZATION PAYBACK

As noted on page 72, equipment costing an estimated \$242,300 would be necessary to facilitate for this work. Payback is estimated at less than one year as detailed below:

Equipment	\$242,300
Installation (30% Est.)	<u>\$ 72,690</u>
Total	\$314,990
Estimated Monthly Savings	\$ 34,354
Payback Period	10 months

#### SUMMARY

This economic analysis shows cost savings in favor of the MoF<sub>2</sub> liner process assuming that the necessary equipment is available and the process of liner preparation is refined. The work accomplished under the MM&T did not achieve the actual yields or production efficiencies upon which this analysis was based. The design of the modified retort and mandrel was governed in part by the physical dimensions of the furnace. The experience gained by these first efforts indicates that the weight of the derby produced could be

increased through a second modification of the transition angle thereby increasing the charge weight but still maintaining liner integrity. Two other areas of importance are the further refinement of the compacting procedure and the positioning of heating coils for the uniform heating of the reduction vessel.

The MM&T successfully demonstrated the feasibility of the process. The questions raised as to liner redesign in terms of transition angle modification, the use of a thinner MgF<sub>2</sub> wall, and the refinement of packing techniques, namely the use of vibrators versus a high frequency jolting table, are matters which we feel could be addressed during the early stages of a start up process. These modifications would be beneficial in achieving the cost savings reflected in the previous section.

## DISCUSSIONS AND CONCLUSIONS

Having completed the program, we will address each of the operations in their respective order.

### MAGNESIUM FLUORIDE POWDER PREPARATION

Due to the abrasive nature of the  $MgF_2$ , the use of a hammer mill to reduce the material to powder form is not recommended. The possibility of iron contamination is present regardless of the hammer material. All of the hammers tried, whether stainless steel, hardened tool steel or those faced with stellite, all exhibited pronounced wear. The most acceptable method, and the one offering the necessary control to yield large amounts of the finer fractions is by ball milling.

### MAGNESIUM FLUORIDE POWDER SIZE AND SHAPE

Through the series of trials where the percentages of the size fractions of the  $MgF_2$  powder were varied, it was apparent that the most dense compact was the result of a blend in which the largest percentage consisted of the finest particle size. In this particular material, size rather than the particle is the more important factor in packing. The best packing conditions were achieved when the energy input was of such a frequency as to permit the movement of the finer particles into the voids, thereby resulting in a smooth hard surface.

### RETORT AND MANDREL DESIGN

A large angular change in the flow direction of the compact does not permit the translation of a uniform packing pressure. The redesign of the retort which resulted in the modified taper as illustrated in the body of the test eliminated this problem.



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## REDUCTION VESSEL PACKING

The jolting action used by the Feed Materials Production Center (NLO) for packing the magnesium fluoride liners could not be duplicated because of a difference in available equipment. However, in our opinion, the use of heavy duty vibrators was very effective for liner formation and are recommended for future use.

## REDUCTION PROVEOUT

Additional work will be required to establish better liner quality in terms of abrasion resistance. The basic procedure of the formation of the liner, through the process of filling with the  $UF_4 + Mg$  blend and finally to the actual reduction reaction, was successful. The derbies selected were processed in accordance with the prescribed specifications and when evaluated, were found to be equal to or slightly better than derbies produced by present methods.

## CHEMISTRY

Chemistry on the proveout derby was equal to that normally produced when the reaction takes place in the presently used graphite liner. The potential for decreasing carbon content is present should the  $MgF_2$  liner be implemented into the production mode. Overall chemistry was within the specifications of MIL-C-63422.

## ULTRASONIC INSPECTION

While the number of the rods subjected to ultrasonic inspection was not overly great, it is nonetheless quite proper to state that the results were impressive. One rod was rejected for a surface defect but there were absolutely no rejects for internal flaws of any kind. Therefore, the quality of the derby metal produced using  $MgF_2$  liners will meet the requirements of all present programs.

## METALLOGRAPHY

Metallographic examination conducted on the extruded rods was much more extensive than normally performed to establish the quality of the metal produced using the  $MgF_2$  liners. There are no indications of any difference in structure when compared to production material. The inclusions were physically smaller and somewhat less in number thereby indicating an improvement in quality.

## SUMMARY

In conclusion, we feel that the results as reported indicate a positive position to the program. There are some additional factors that deserve to be addressed in the area of refinement of design of the reduction vessel and other performance parameters. However, in our opinion, these are not areas to present major concern.

In view of the overall results of the program with its obvious potential to improve derby chemistry, it is recommended that those specific areas such as liner compaction and temperature control be addressed as part of a general start up procedure. In our opinion, these are basically engineering problems for which there is already some data available generated under this MM&T program which could be useful as a starting point in resolution of those problems.

**TABLES**

TABLE 1

Mg F <sub>2</sub> POWDER COMPACTION TESTS					
In Arbitrary Dieter - Detroit Hardness Units					
MATERIAL	Tapped as Packed	Pressure lbs./ sq. in.			
		800	1600	2400	3200
FM PC Trial Powder	72	72	78	78	80
	66	75	78	80	81
	70	67	70	75	83
	53	78	81	83	77
	75	64	75	74	78
Average	67.2	71.2	76.4	77.8	79.8
CMI No 2.1 -325 = 40% +325 - 200 = 25% +200 - 65 = 25% +68 = 10%	40	50	64	68	73
	45	53	60	72	75
	51	48	63	70	71
	47	55	67	72	76
	40	51	60	70	74
Average	44.6	51.4	62.8	70.4	73.8
CMI No 2.2 -325 = 50% +325 - 200 = 30% +200 - 65 = 15% + 68 = 5%	45	70	80	78	82
	41	63	72	77	81
	52	66	74	78	78
	47	71	75	79	81
	45	69	75	78	80
Average	46.0	67.8	75.2	78.0	80.4
CMI No 2.3 -325 = 55% +325 - 200 = 30% +200 - 65 = 10%	40	75	75	82	82
	45	71	76	80	81
	47	71	78	81	81
	43	72	76	81	83
	49	76	80	78	82
Average	44.8	73.0	77.0	80.4	81.8
CMI No 2.4 -325 = 68% +325 - 200 = 25% +200 - 65 = 10%	45	78	83	86	89
	50	74	80	84	87
	47	76	81	82	86
	54	76	82	82	86
	51	72	81	83	86
Average	49.4	75.2	81.4	83.4	84.0

TABLE 2

## LINER PACKING TESTS

Test No	MgF <sub>2</sub> Powder	Retort	Mandrel	Packing Method	Result
1	CMI Mix A	Std. CMI	Std. CMI	Jolt 30 min. at 50/min. + 25 min. at 120/min.	Transition cave in
2	CMI Mix A	Std. CMI	Std. CMI	Jolt 6 min. at 25/min. Refill +4 min. at 25/min.	Cave in hardness 65 small wall section
3	CMI Mix B	Std. CMI	Std. CMI	Jolt 6 min. at 25/min. Refill +4 min. at 25/min.	Cave in
4	CMI Mix A	Std. CMI	Std. CMI	Jolt 22 min. at 50/min. + 28 min. at 150/min.	Top hardness 75 cave in hardness 65 on remaining wall
5	CMI Mix B	Std. CMI	Std. CMI	Jolt & Vibrator 30 min. at 50/min. Refill+27 min. at 50/min. + 25 min. at 120/min.	Transition cave in
6	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Jolt 15 min. at 50/min. + 30 min. at 120/min.	Cave in

TABLE 2 (Cont'd.)

## LINER PACKING TESTS

Test No	MgF <sub>2</sub> Powder	Retort	Mandrel	Packing Method	Result
7	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Jolt 15 min. at 50/min. → 30 min. at 120/min.	Cave in
8	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Vibration, base plate 30 min.	Partial cave in. Hardness 65
9	FMPC	Straight Wall	Long taper Thick wall	Vibration, base plate 30 min.	Good hardness 78
10	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	1. Fill to transition jolt 100x 2. Fill 3/4 jolt 100x 3. Fill to top vibrate 30 min.	Cave in at transition
11	CMI Mix A	Straight Wall	Long taper Thick wall	Vibration, base plate 30 min.	Good
12	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Vibration, base plate 30 min. Fill - vibrate 30 min.	Cave in

TABLE 2 (Cont'd.)

## LINER PACKING TESTS

Test No	Mgf 2 Powder	Retort	Mandrel	Packing Method	Result
14	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Vibration, base plate 20 min. fill - vibrate 40 min.	Cave in
15	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Jolted & vibrated same time 20 min. at 50/min. + 20 min. at 120/min.	Cave in
16	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Jolt only 30 min. at 50/min. + 30 min. at 120/min.	Cave in
17	CMI Mix A	1/4 scale Std. CMI	1/4 scale Std. CMI	Vibration, base plate 20 min. fill - vibrate 40 min.	Cave in
18	CMI Mix A	1/4 scale Std. CMI	1/4 scale Std. CMI	Jolted & vibrated same time 20 min. at 50/min. + 20 min. at 120/min.	Cave in
19	CMI Mix A	1/4 scale Std. CMI	1/4 scale Std. CMI	Jolt only 30 min. at 50/min. + 30 min. at 120/min.	Cave in
20	FMPC	1/4 scale Std. CMI	1/4 scale Std. CMI	Jolt 5 min. at 60/min. fill 30 min. 60/min.	Failed at bottom and transition



TABLE 2 (Cont'd.)

## LINER PACKING TESTS

Test No	Mgf 2 Powder	Retort	Mandrel	Packing Method	Result
21	CMI Mix A	1/4 scale Std. CMI	1/4 scale Mod. taper	Vibration, base plate 30 min.	Side good failed at bottom
22	CMI Mix A	1/4 scale Std. CMI	1/4 scale Std. CMI	Vibration, base plate 45 min.	Good liner
23	CMI Mix A	1/4 scale Std. CMI	1/4 scale Mod. taper	Vibrate 1 min. - measure drop + 5 min. - measure drop +10 min. - measure drop Special packing test.	Insufficient Packing time failed
24	CMI Mix A	1/4 scale Std. CMI	1/4 scale Mod. taper	Vibrate 1 min., fill + 1 min., fill +30 min.	Good No breaks
25	FMPC	1/4 scale Std. CMI	1/4 scale Mod. taper	Fill bottom 1/2 " Vibrate 5 min. fill measure Drop +30 min.	Good No breaks
26	FMPC	1/4 scale Mod. taper	1/4 scale Mod. taper	Vibration, base plate 45 min.	Good Smooth surface

TABLE 2 (Cont'd.)

## LINER PACKING TESTS

Test No	Mg <sub>2</sub> Powder	Retort	Mandrel	Packing Method	Result
27	CMI Mix A	1/4 scale Mod. taper	1/4 scale Mod. taper	Vibration, base plate 45 min.	Good Smooth surface
28	CMI Mix A	1/4 scale Std. CMI	1/4 scale Std. CMI	Vibration, base plate 45 min.	Transition Cave in
29	CMI Mix A	1/4 scale Mod. taper	1/4 scale Mod. taper	Vibration, base plate 30 min.	Good
30	CMI Mix A	Mod. taper	Mod. taper	3 Vibrators 120° apart Fill lower section vibrate 1 hr. Fill mid section vibrate 1 hr. Fill top section vibrate 1 hr.	Small crack at bottom Surface Smooth
31	CMI Mix A	Mod. taper	Mod. taper	3 Vibrators 120° apart Same	Good
32	CMI Mix A	Std. CMI	Std. CMI	3 Vibrators 120° apart Same	Transition Cave in
33	CMI Mix A	Mod. taper	Mod. taper	3 Vibrators 120° apart Fill lower section vibrate 1 hr. Fill mid section vibrate 1 hr. Fill top section vibrate 1 hr.	Good Smooth surface
34	CMI	Std. CMI	Mod. taper	3 Vibrators 120° apart Fill lower section vibrate 1 hr. Fill mid section vibrate 1 hr. Fill top section vibrate 1 hr.	Good Smooth surface

TABLE 3

## REDUCTION TEST LINERS

Test No	MoF <sub>2</sub> Powder	Retort	Mandrel	Packing Method	Result
X-1	CMI Mix A	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good Smooth surface
X-2	CMI Mix A	Std. CMI	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Generally good. Minor break at thick transition section.
X-3	CMI Mix B	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good. Slight rough edge at transition
X-4	CMI Mix A	Std. CMI	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good. Minor break of thick transition section.
X-5	CMI Mix B	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good Clean edges

TABLE 3.

## REDUCTION TEST LINERS (Cont.d.)

Test No	Mg <sub>2</sub> Powder	Retort	Mandrel	Packing Method	Result
X-6	CMI Mix A	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good. Minor edge break at transition.
X-7	CMI Mix A	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good. Alignment better. No edge break
X-8	CMI Mix B	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good. Smooth surface - minor break at transition.
X-9	CMI Mix A	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good. Smooth surface - minor break at transition.
X-10	CMI Mix B	Mod. taper	Mod. taper	Vibration, 3 units 120° Fill lower section - 1 hr. Fill mid section - 1 hr. Fill top section - 1 hr.	Good. Smooth surface. No edge break at transition.

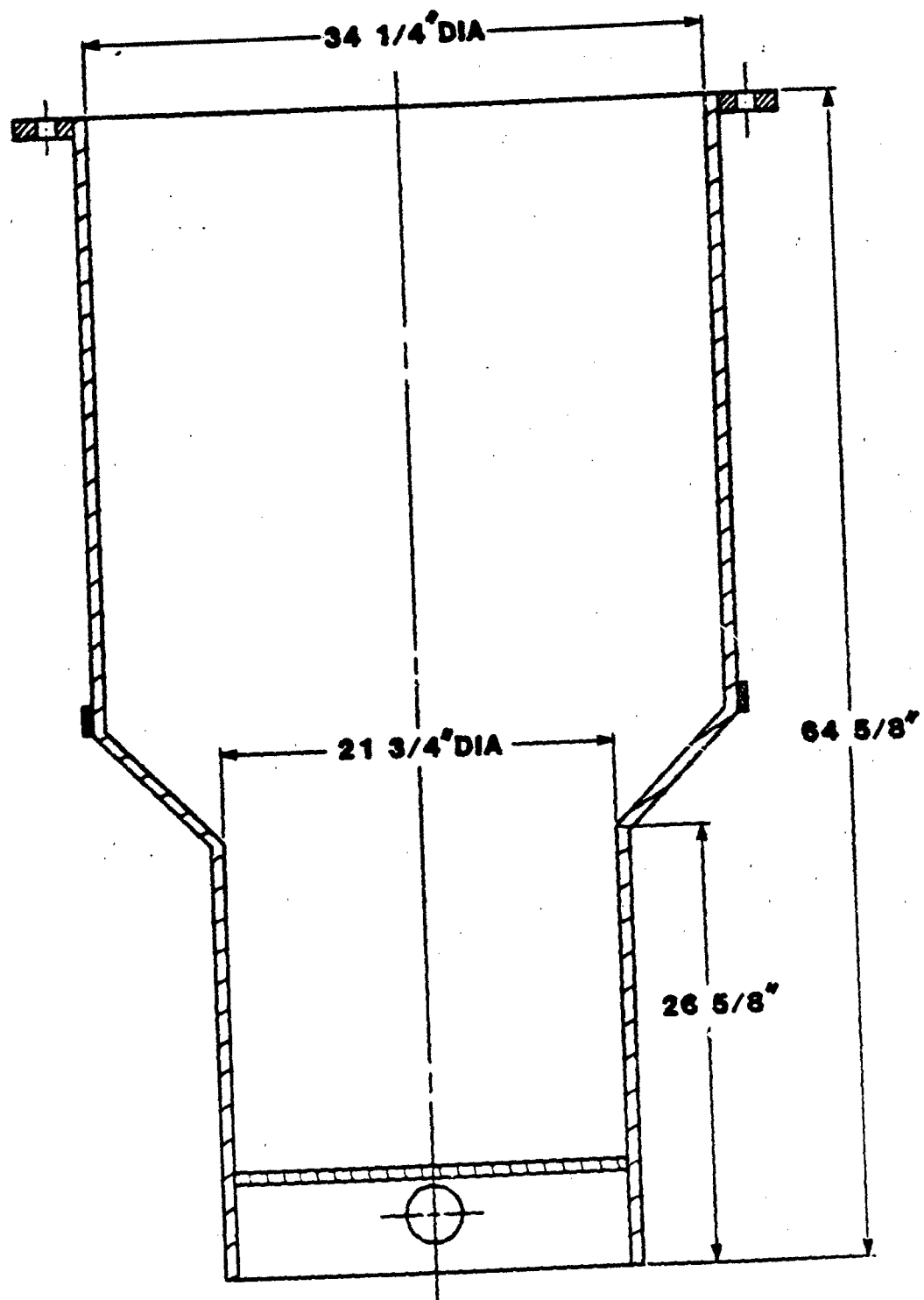
TABLE 4

## PRODUCTION PROVE-OUT CHEMISTRY

Sample No.	Element Determination								
	Ti (wt%)	C (wt%)	Fe	Ni	Cu	Si	Mg	F	
X-7-1-1	0.73	0.005	14	4	1	51	2	8	
X-7-1-10	0.73	0.005	14	4	1	52	4	4	
X-8-5-1	0.73	0.005	19	4	1	52	3	4	
X-8-5-11	0.73	0.007	22	5	3	47	3	6	
X-10-1-1	0.71	0.006	38	10	2	46	1	4	
X-10-1-10	0.73	0.007	36	9	2	44	1	4	

NFI Ref. No. QC-893

**FIGURES**



**FIG. 1A) STEEL RETORT AS USED AT C.M.I.**

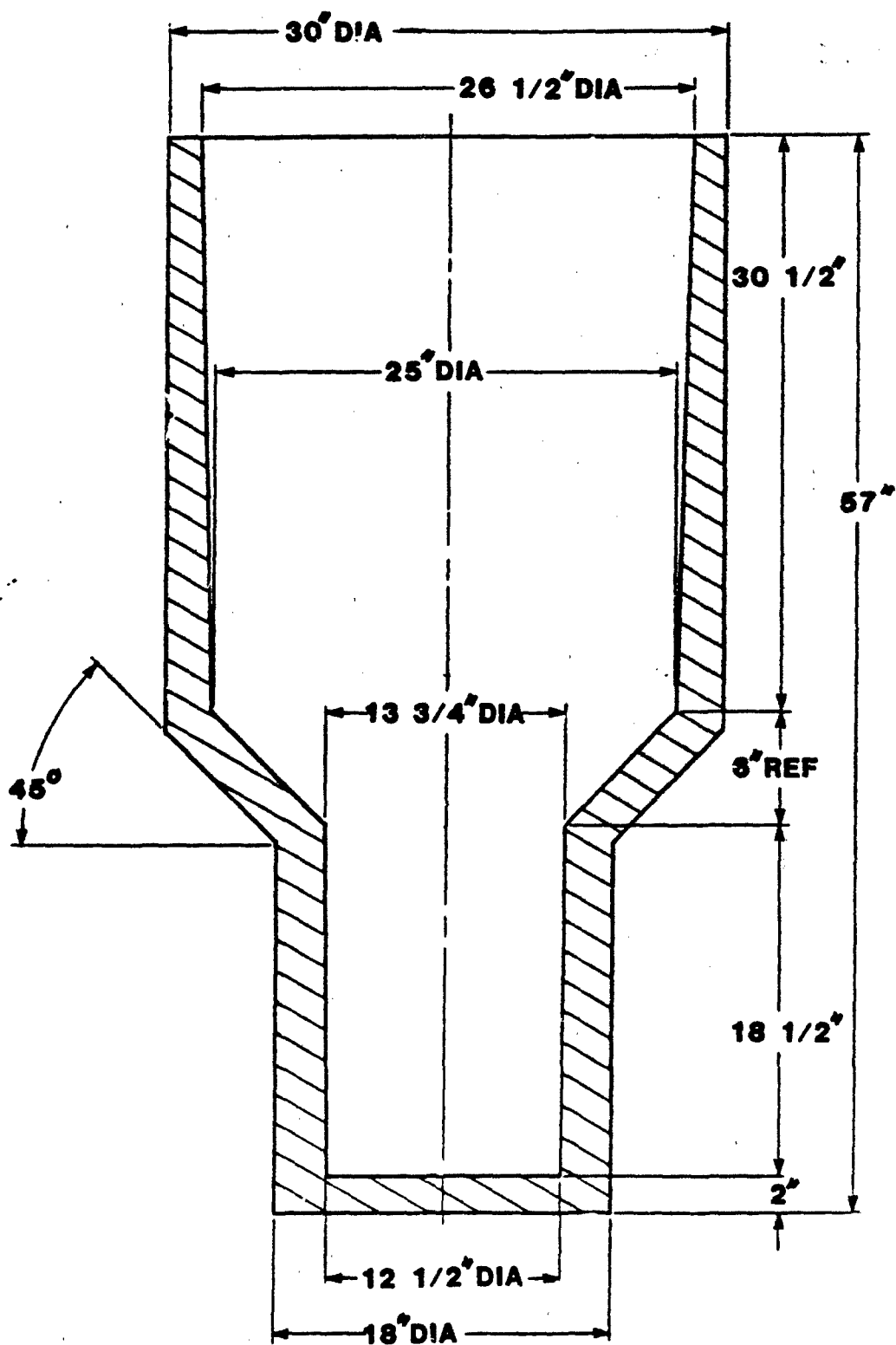
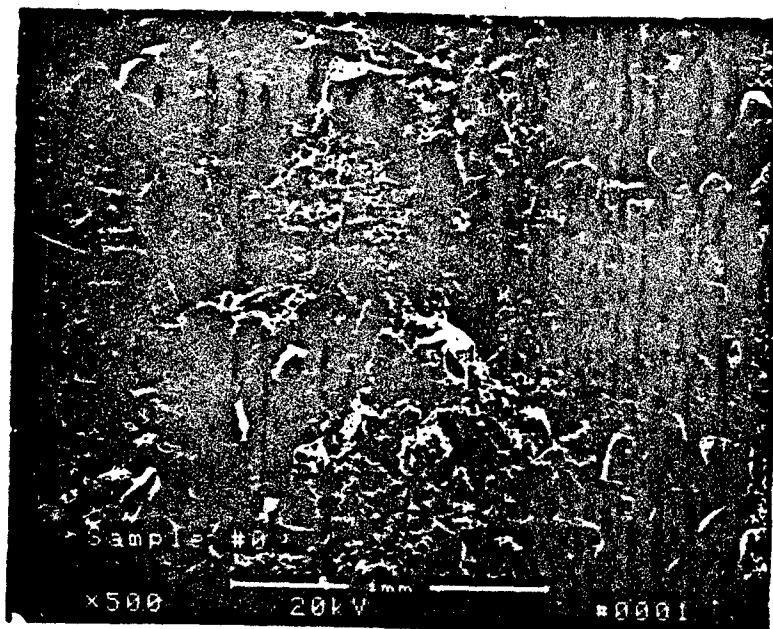


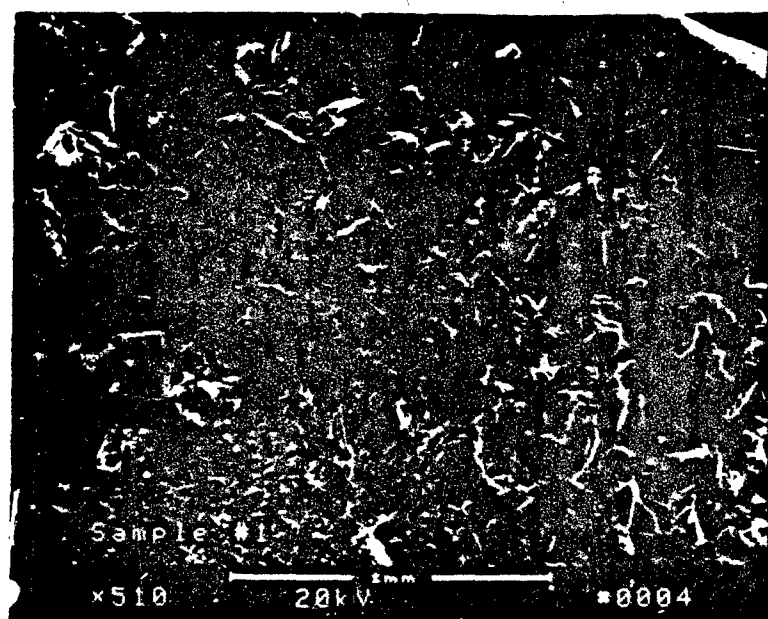
FIG. 1B) GRAPHITE LINER AS USED AT C.M.I.





FMPC

Mag. 50 x



CMI

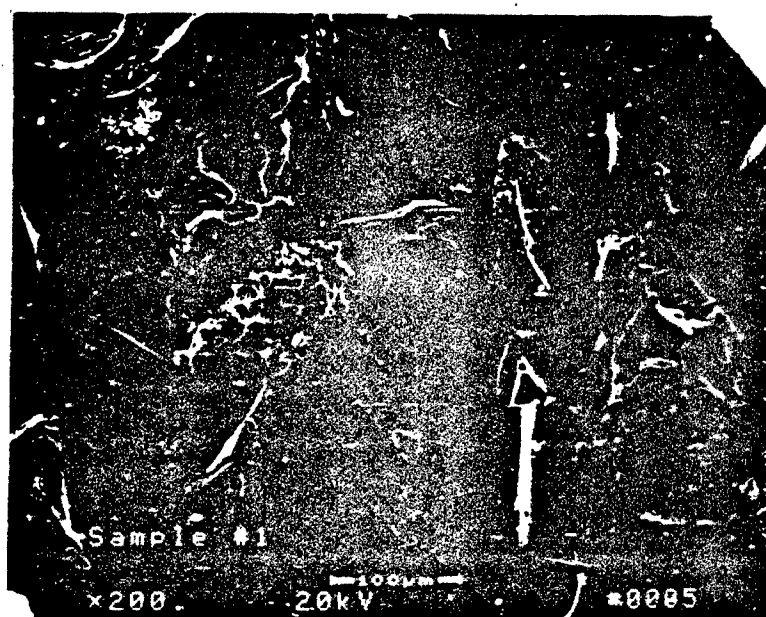
Mag. 50 x

Figure 2. Scanning Electron Microscopy Photographs of  $\text{MgF}_2$  Particles



FMPC

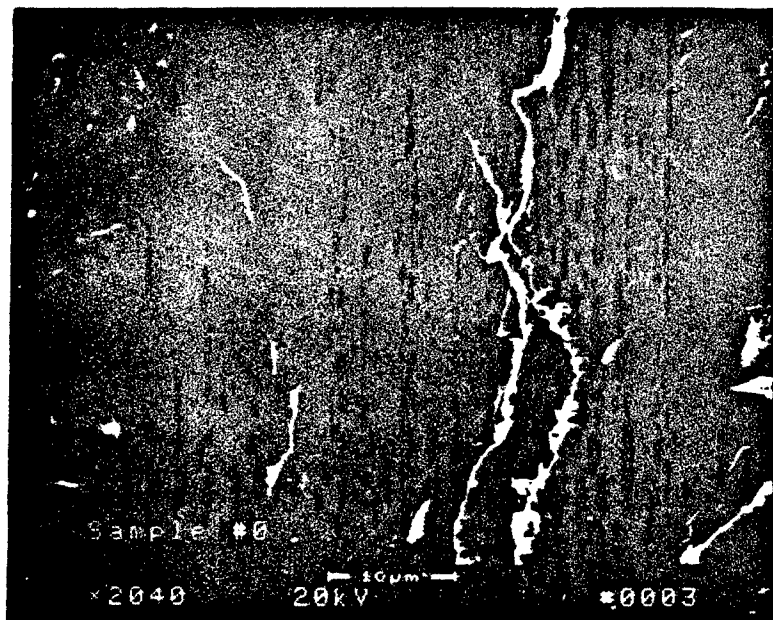
Mag. 200 x



CM1

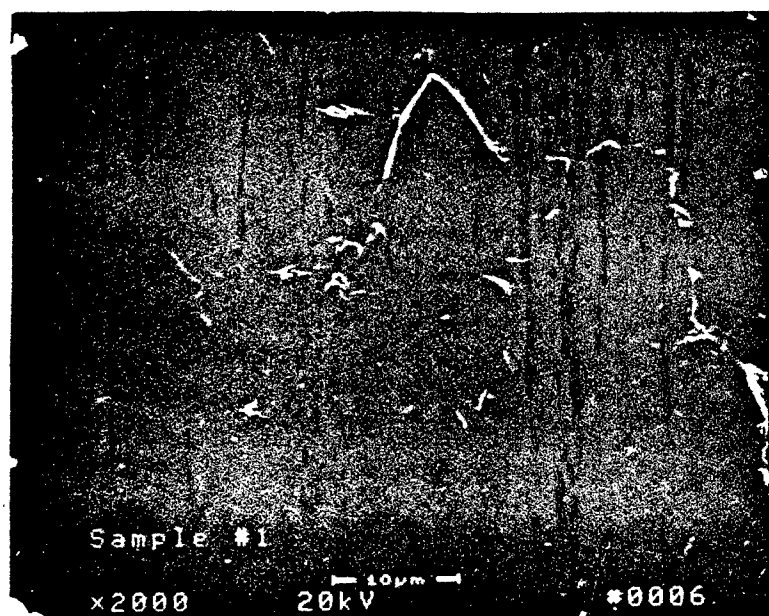
Mag. 200 x

Figure 3. Scanning Electron Microscopy Photographs of  $MgF_2$  Particles



FMPC

Mag. 2000 x



CMI

Mag. 2000 x

Figure 4. Scanning Electron Microscopy Photographs of  $MgF_2$  Particles



Figure 5. Hammer Mill

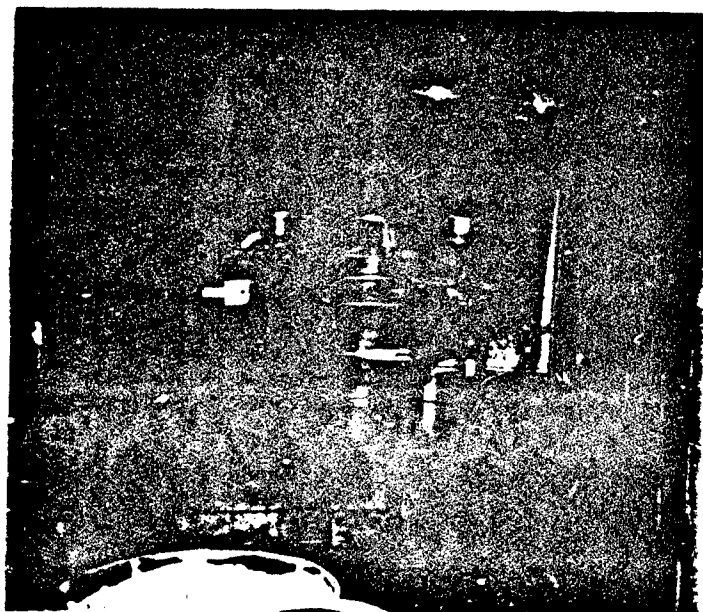


Figure 6. Sweco Classifier



Figure 7. Laboratory Packing Press

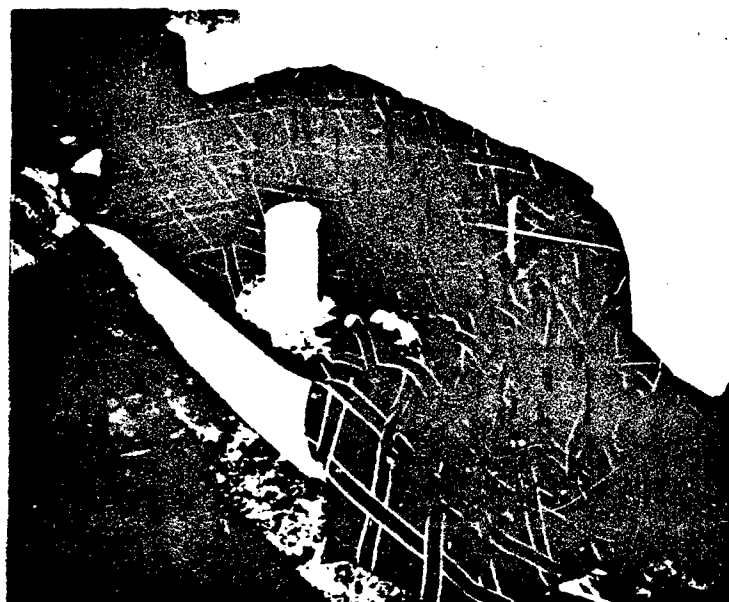


Figure 8.  $\text{MgF}_2$  Core

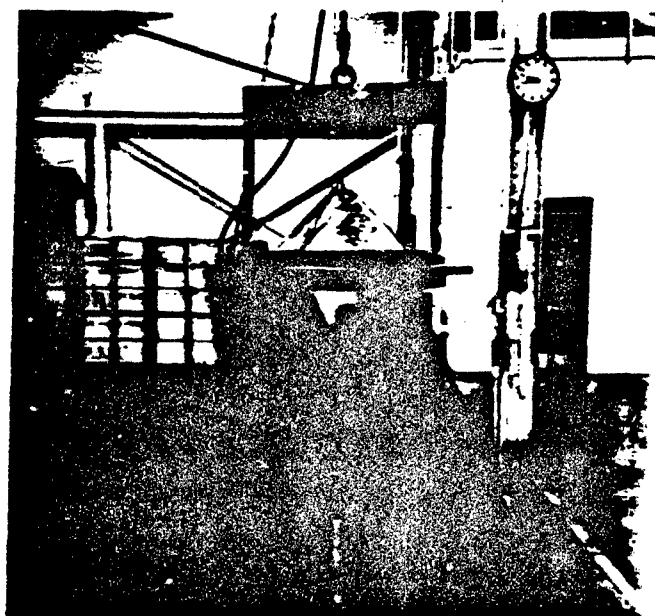


Figure 9. Standard Mandrel

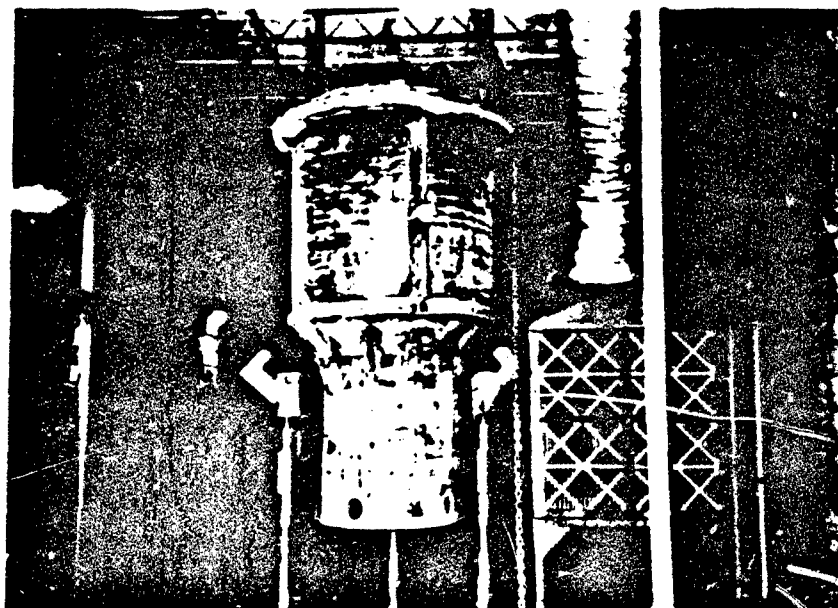


Figure 10. Jolter Table Tie Down

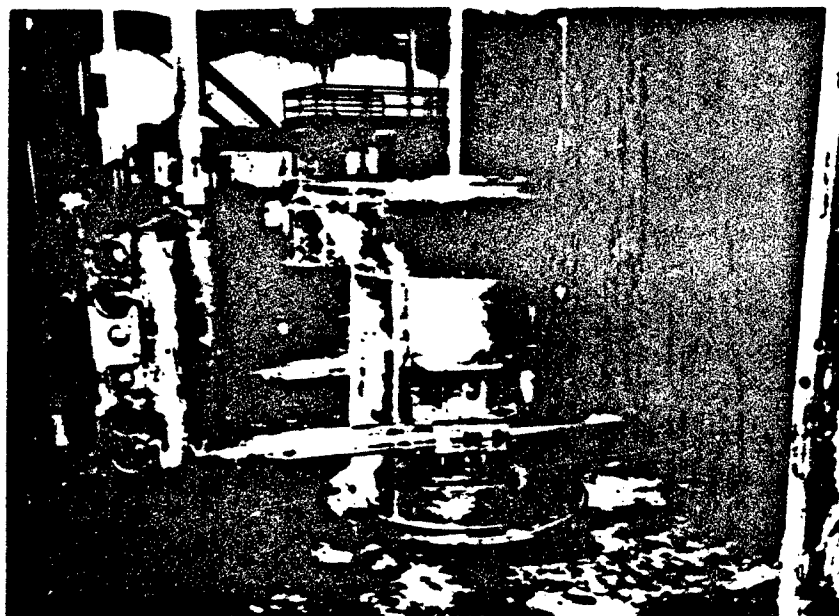
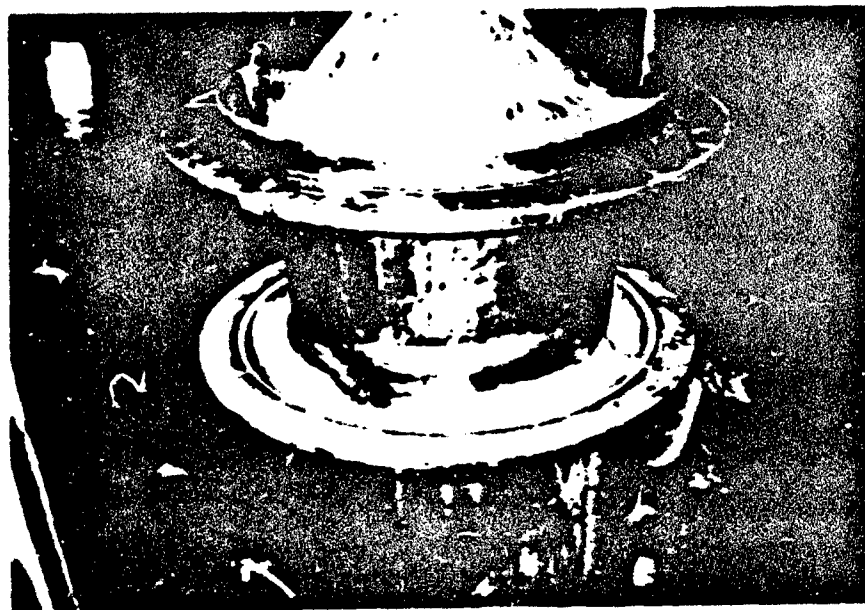


Figure 11. Hopper Filler



Figure 12. Formed Liner



Figures 13A and 13B. Standard Mandrel Sidewall Collapse





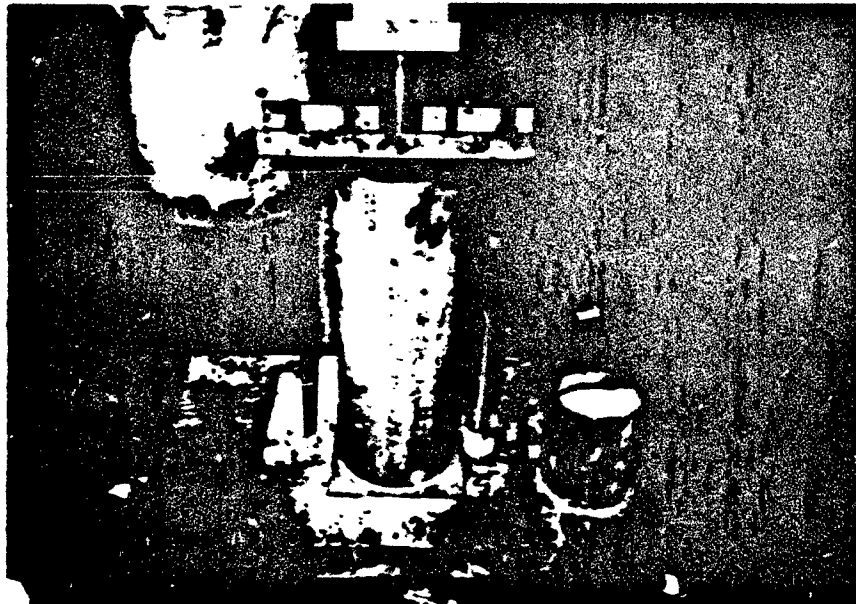


Figure 14. Taper Mandrel Test Unit



Figure 15. Mandrel Withdrawal

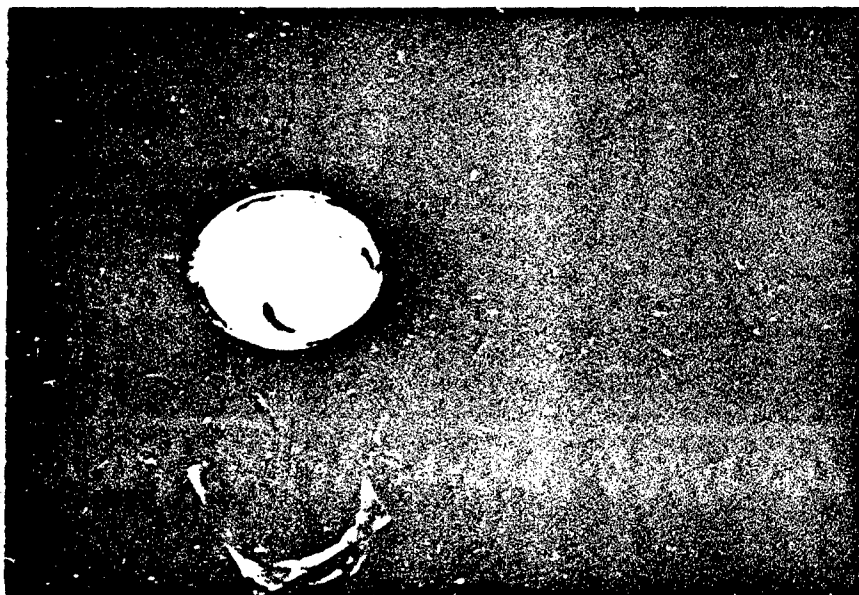


Figure 16. Formed Core

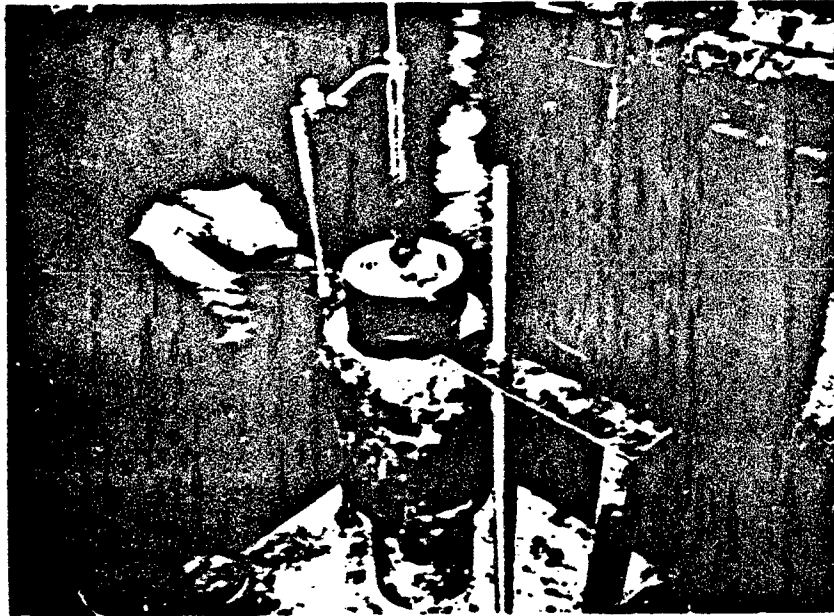


Figure 17. 1/4 Scale Standard Mandrel Withdrawal

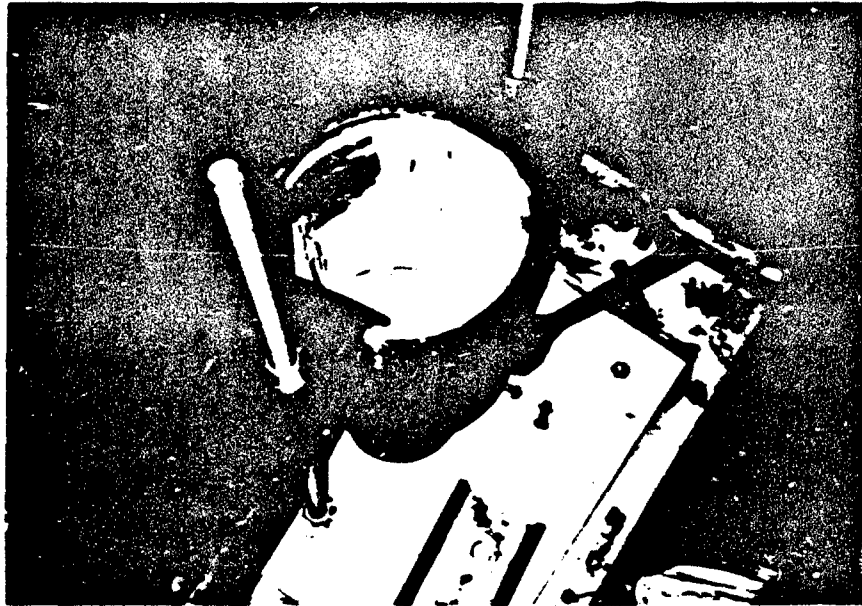


Figure 18. Standard Mandrel Collapse

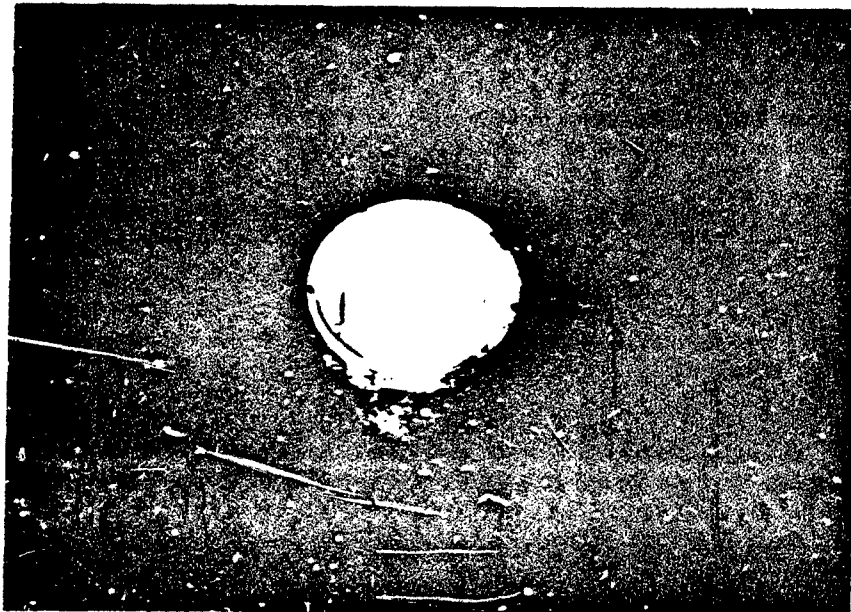


Figure 19. Standard Mandrel Collapse

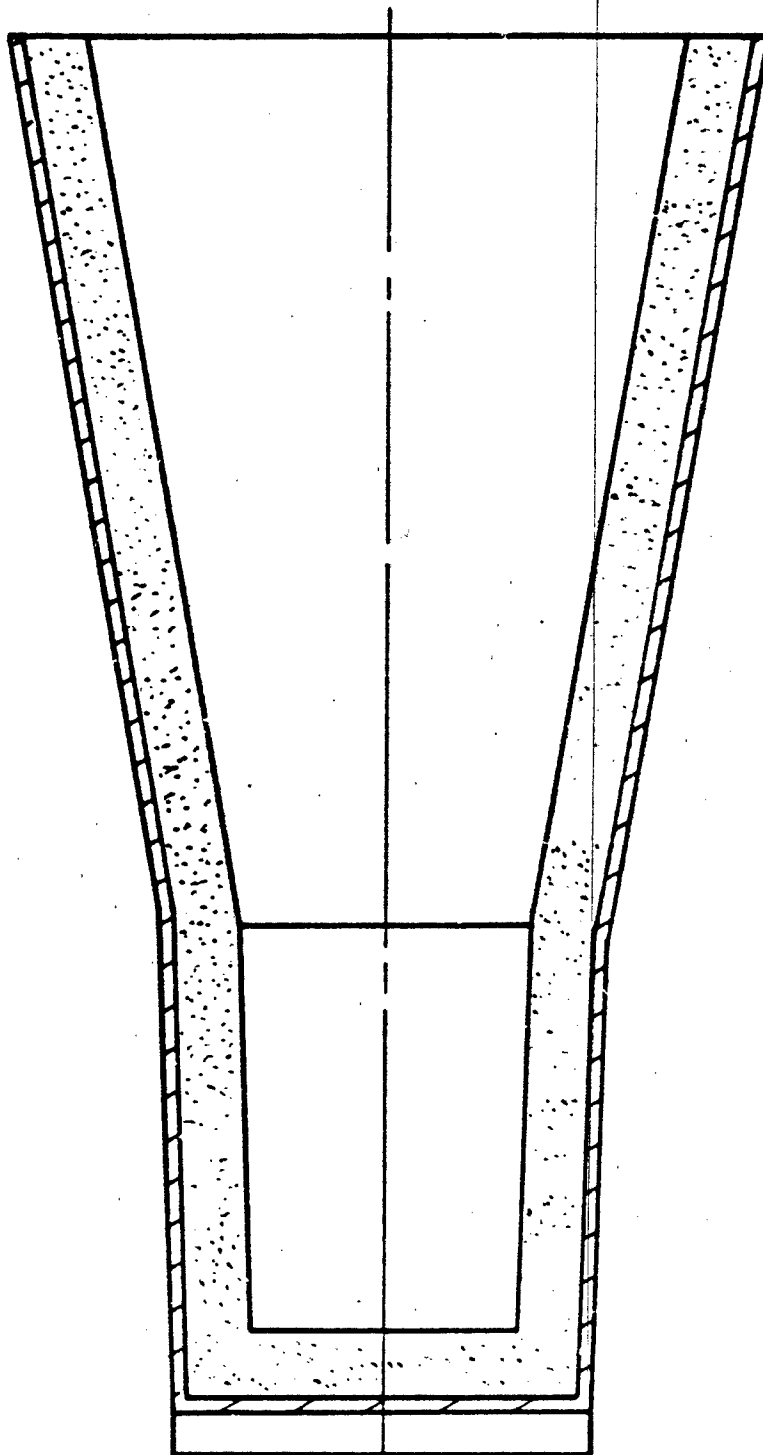


Figure 20. Modified Taper Design

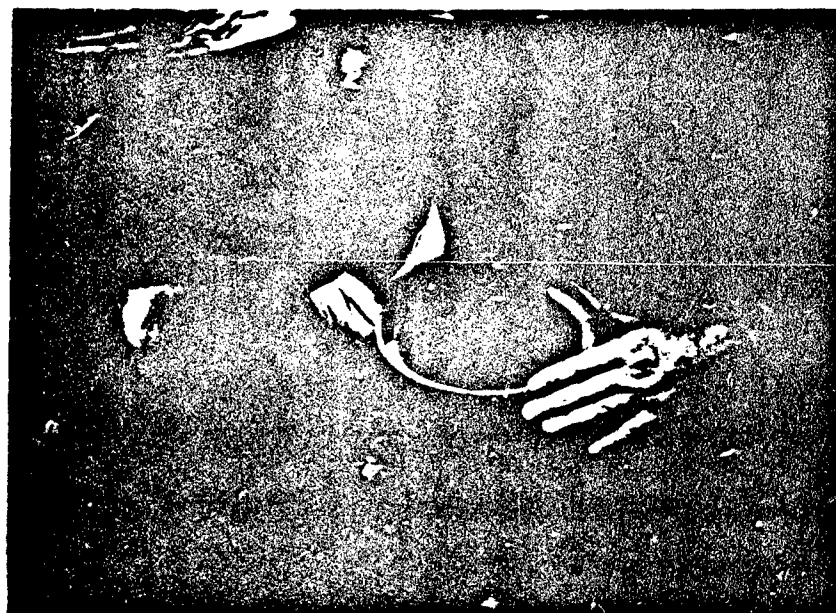


Figure 21. Taper Liner Withdrawal 1/4 Scale



Figure 22. Formed Taper Liner 1/4 Scale

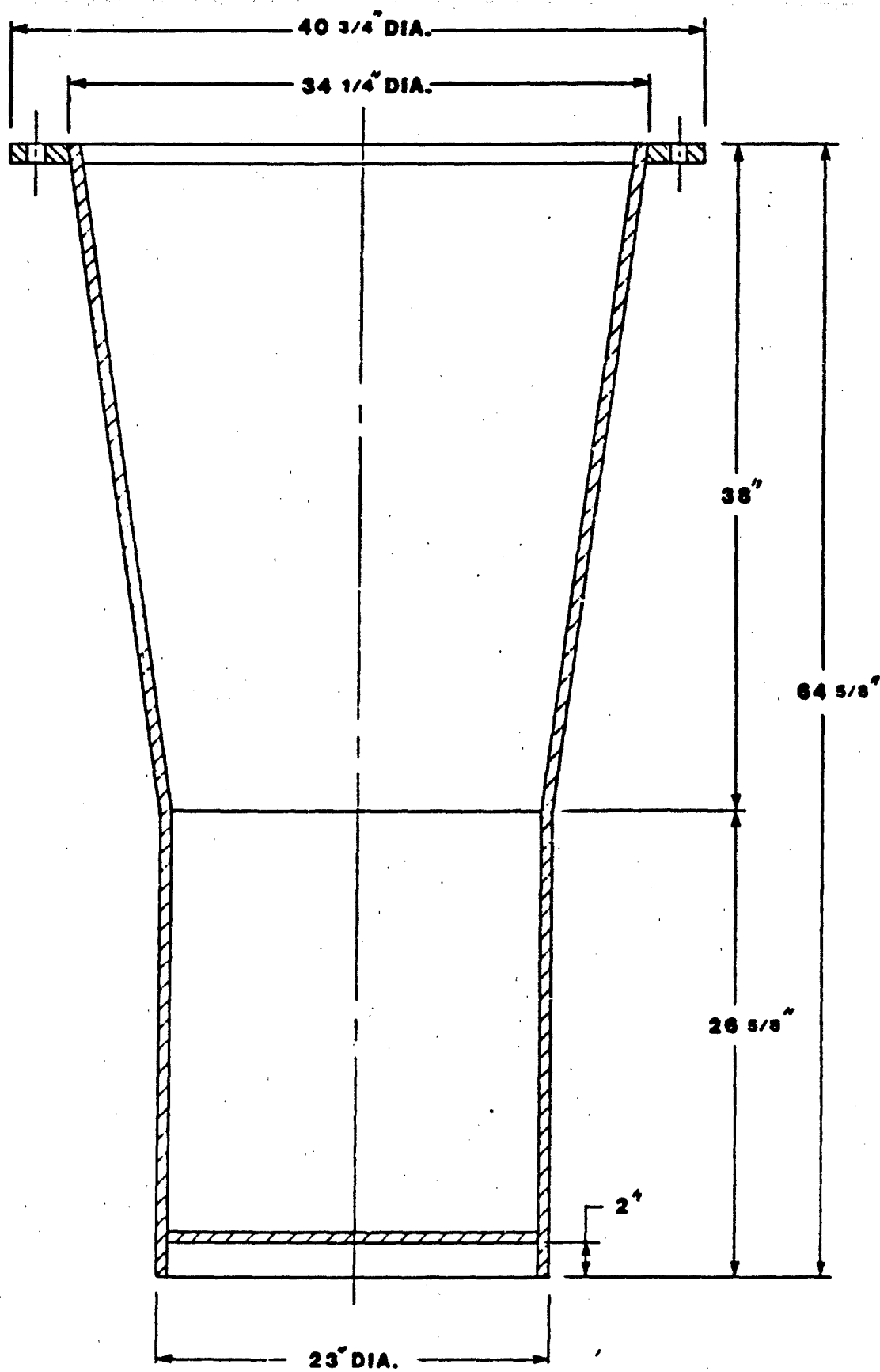


Figure 23. Modified Taper Retort

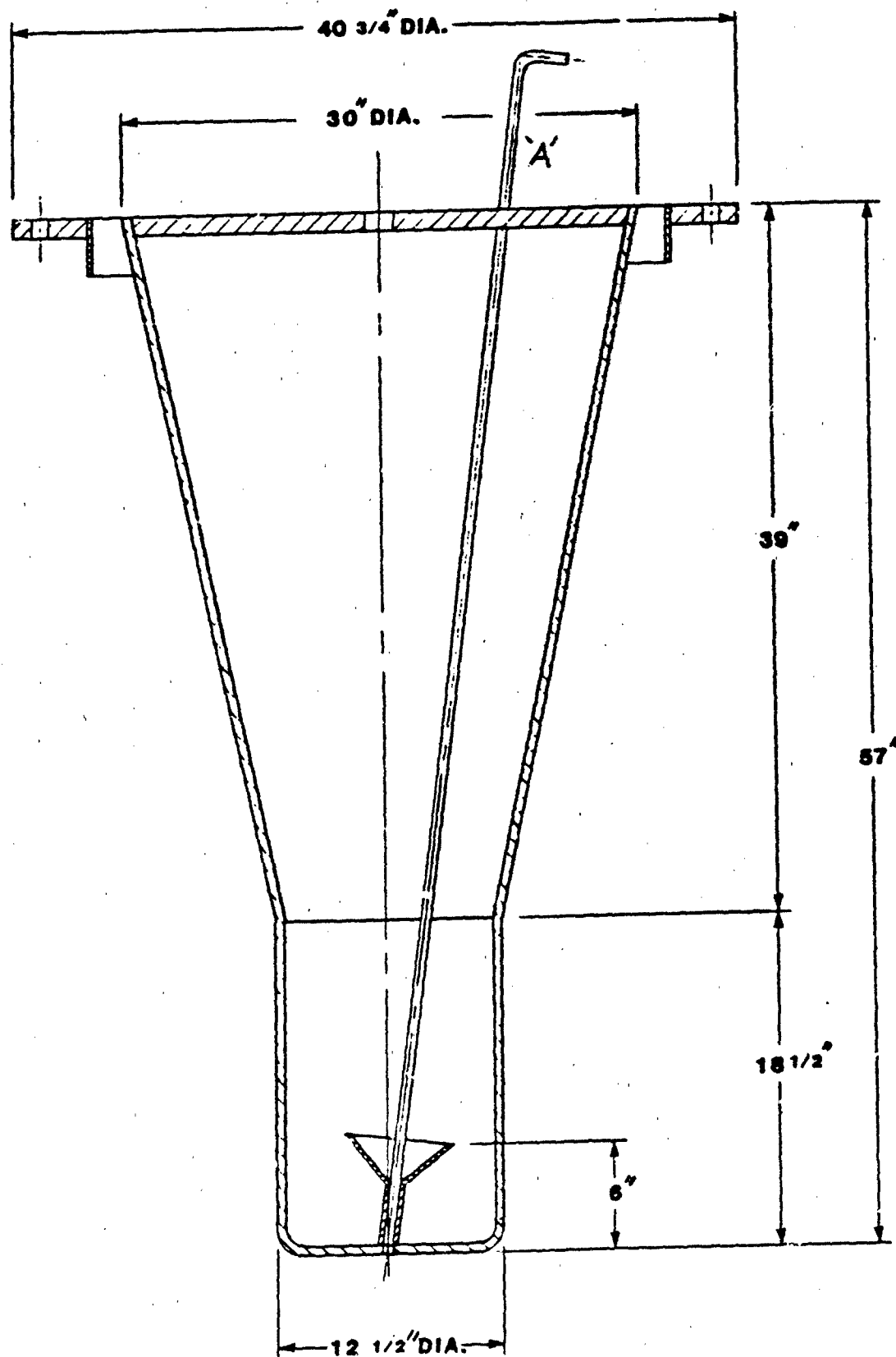


Figure 24. Modified Taper Mandrel



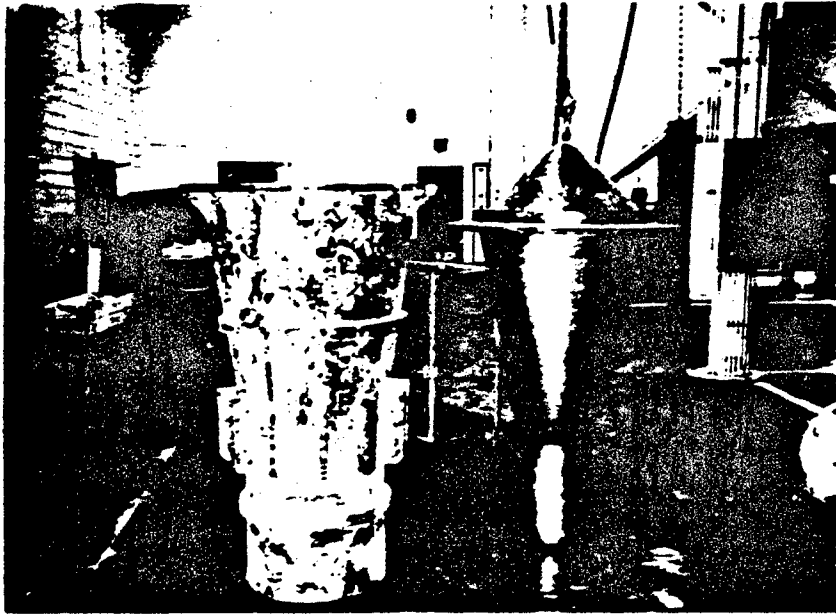


Figure 25. Modified Taper Retort & Mandrel

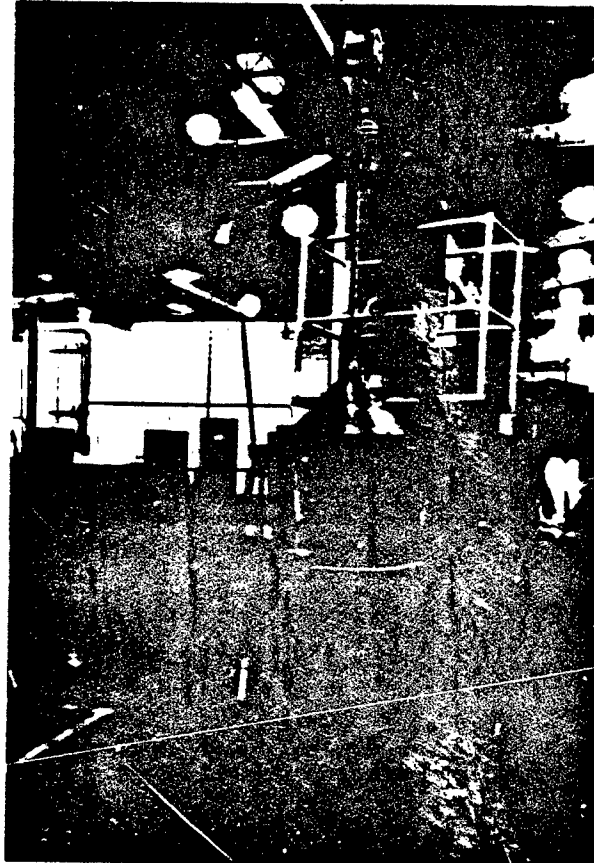


Figure 26. Mandrel Removal



Figure 27. Partial Withdrawal of Taper Mandrel

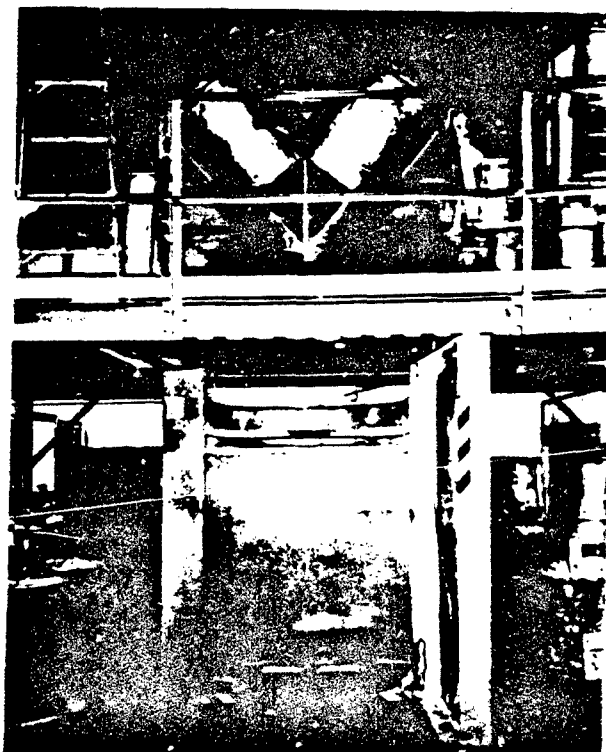


Figure 28. Blending Station



Figure 29. Attachment of Snorkle Tube

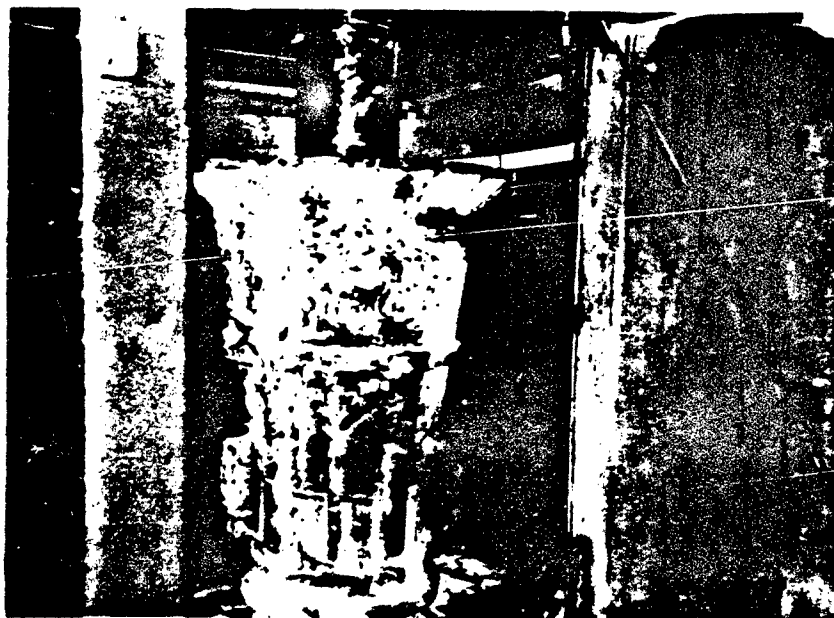


Figure 30. Vessel in Fill Position

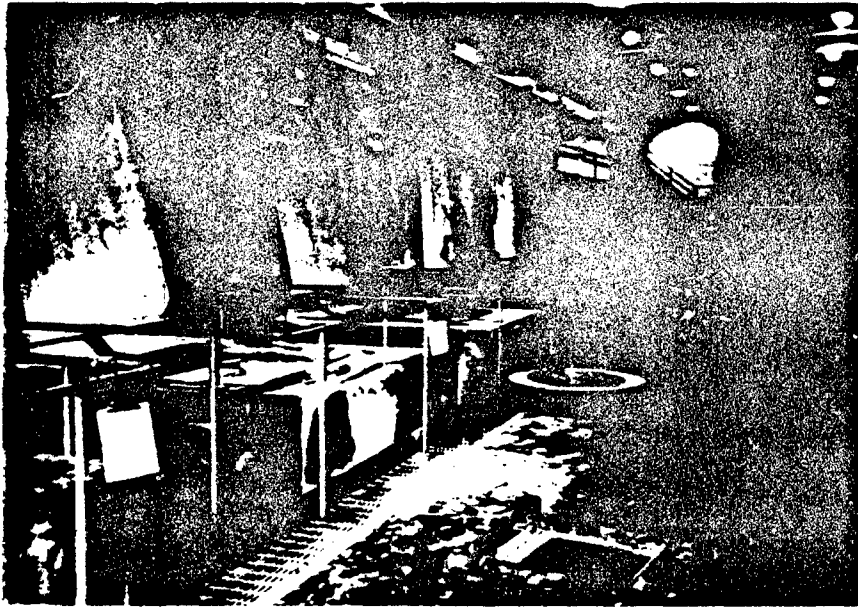


Figure 31. CMI Furnace Line

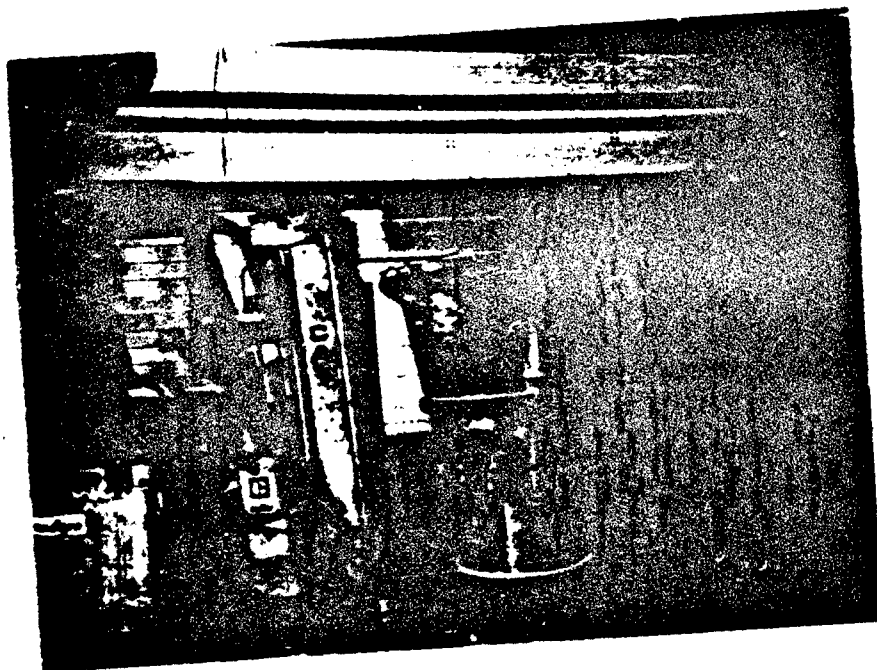


Figure 32. Vessel Transport

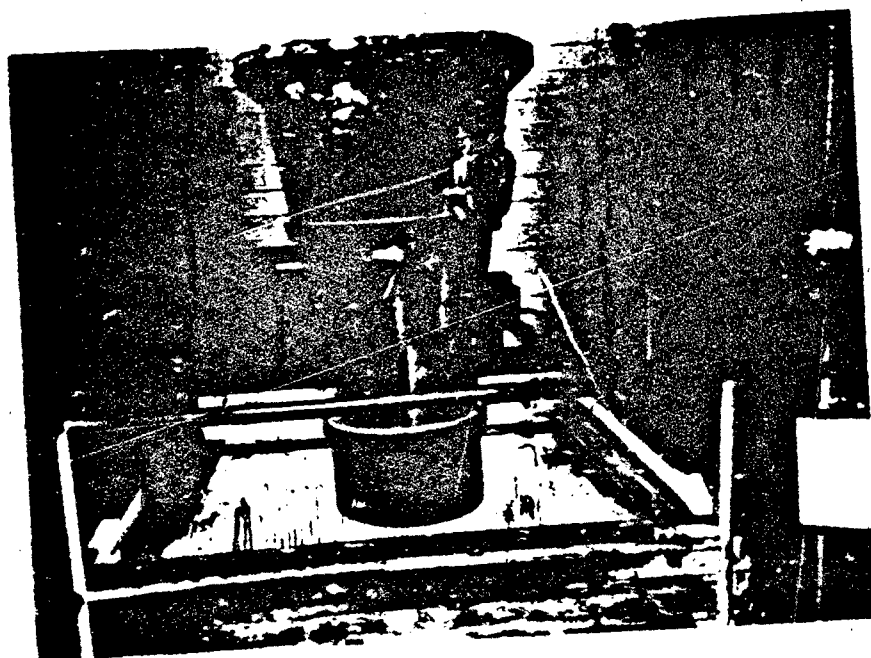


Figure 33. Overhead Handling



Figure 34. Retort in Pit Furnace

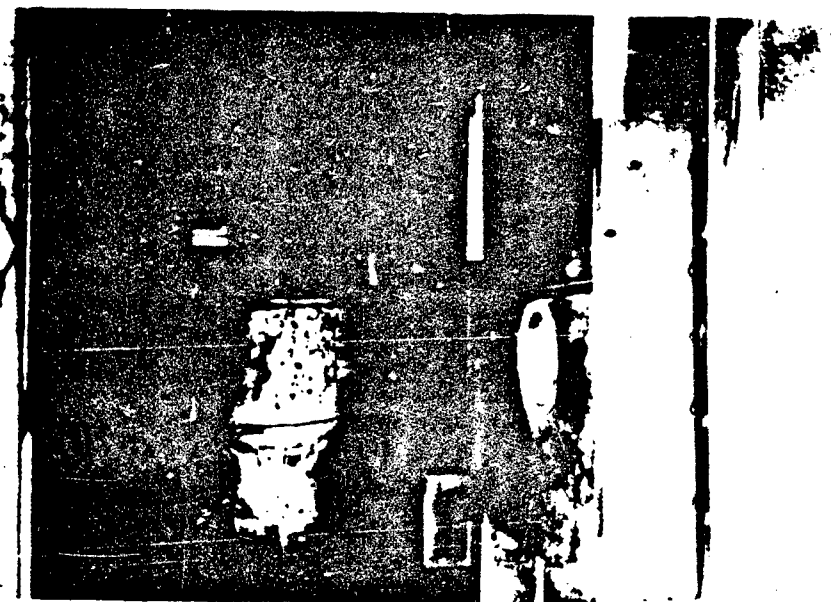


Figure 35. Cooling Chamber

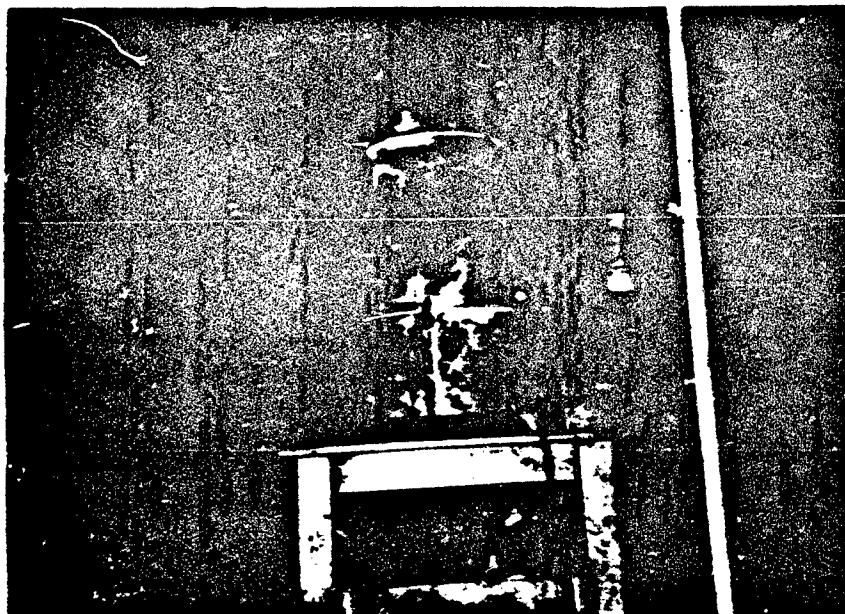


Figure 36. Knockout Station

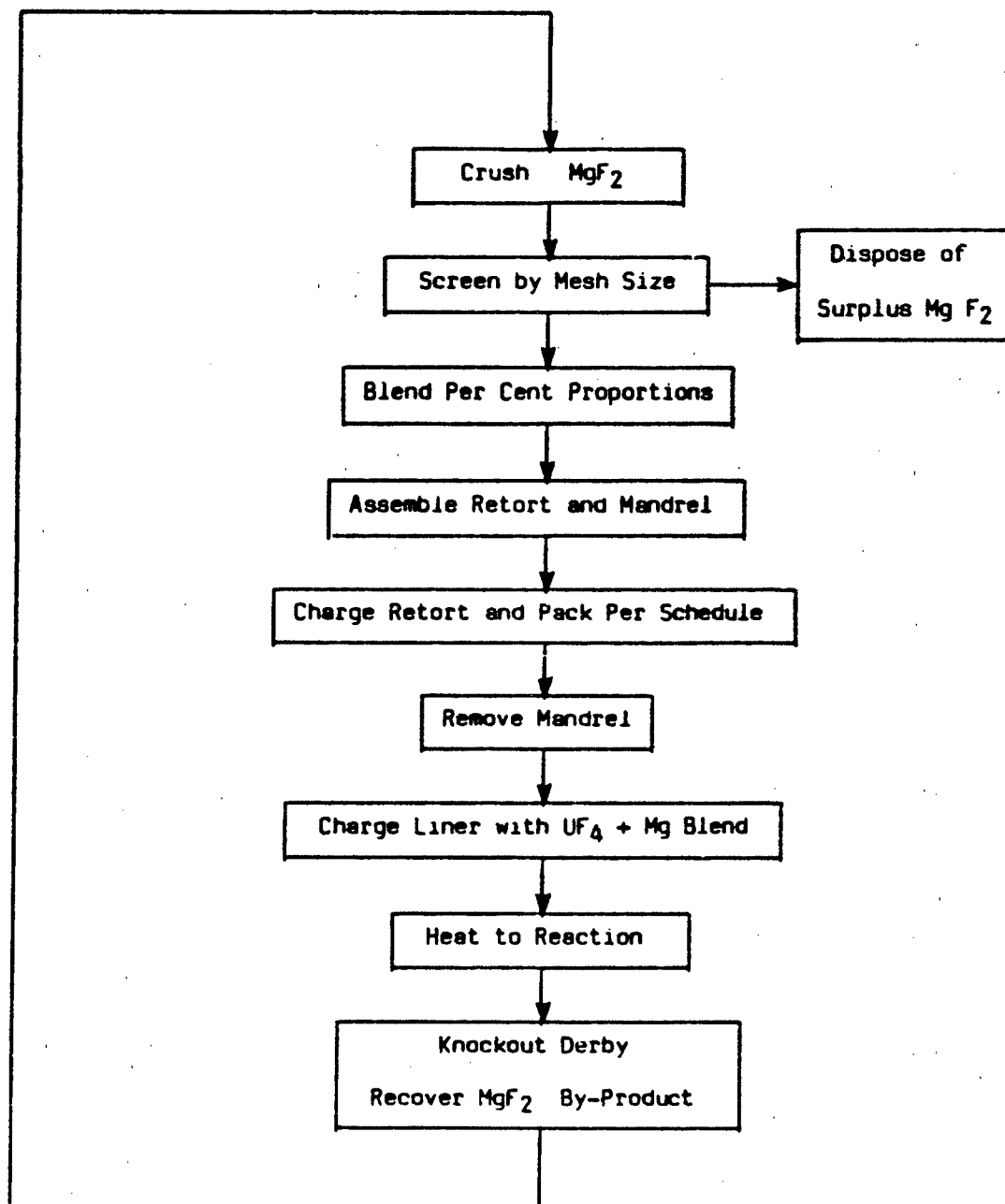
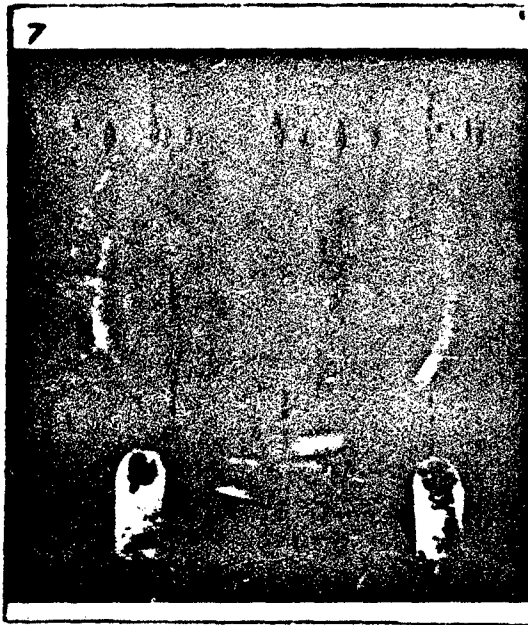


Figure 37. Mg F<sub>2</sub> Liner Derby Flow Sheet

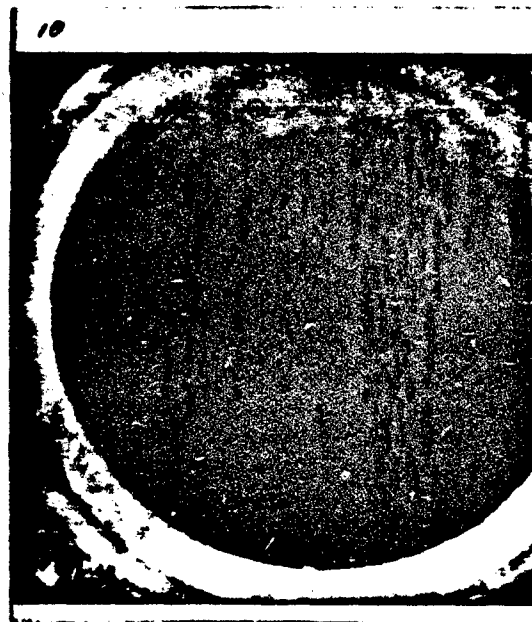




Liner x-7



Liner x-8



Liner x-10

Figure 38. Proveout Production Liners

Production prove-out of  $MgF_2$  liner produced derbies was carried out on 3 derbies according to Process Control Document No. 833-051 operations No. 1 through No.13 as illustrated by the flow chart as follows:

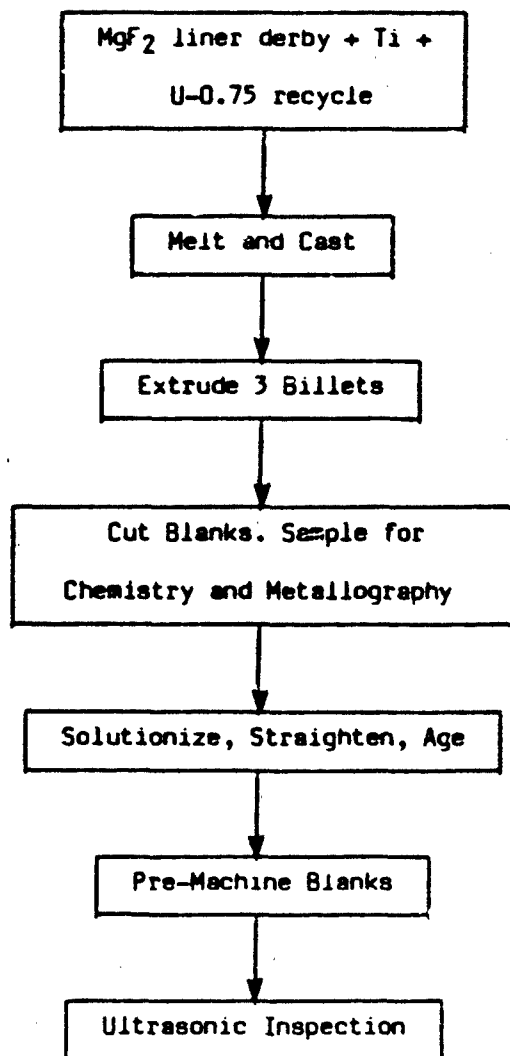


Figure 39. Production Proveout Flow Sheet



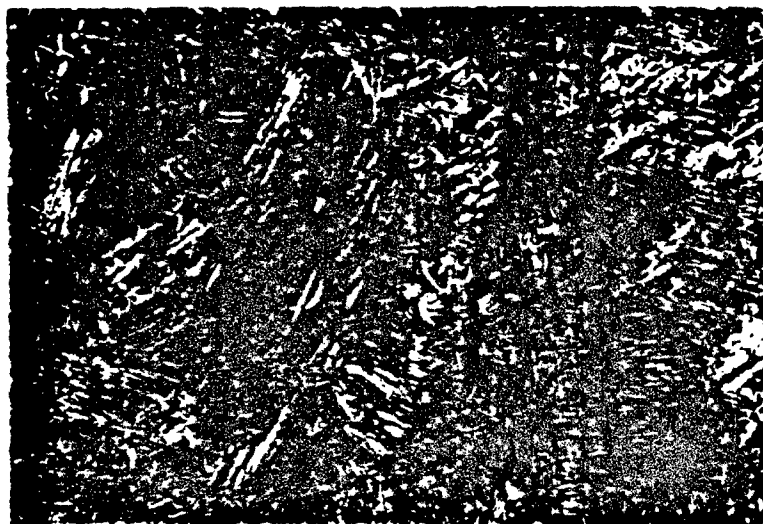
Edge

50x



Mid-radius

50x



Center

50x

X-7-1-1

Figure 40. Photomicrographs of Proveout Derby Extrusions



Edge

100x



Mid-radius

100x



Center

100x

X-7-1-1

Figure 41. Photomicrographs of Proveout Derby Extrusions



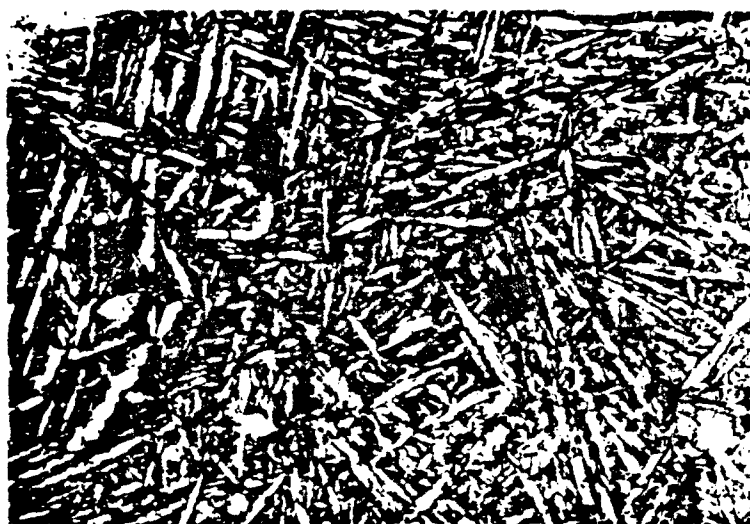
Edge

250x



Mid-radius

250x



Center

250x

X-7-1-1

Figure 42. Photomicrographs of Proveout Derby Extrusions



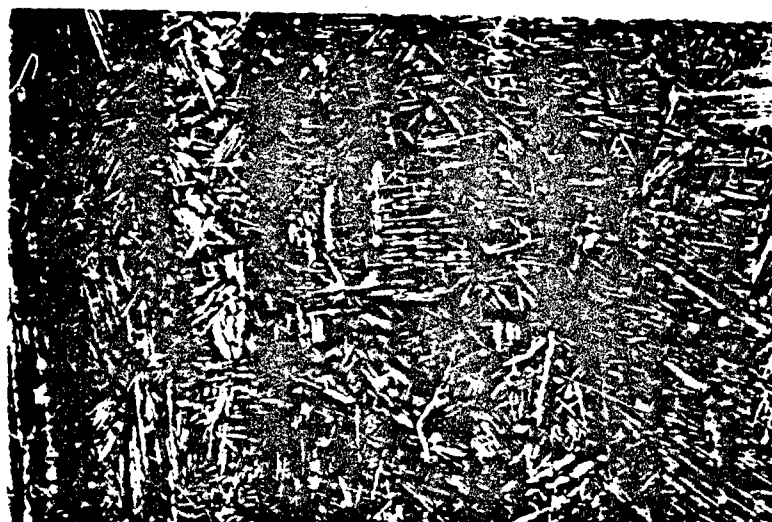
Edge

50x



Mid-radius

50x

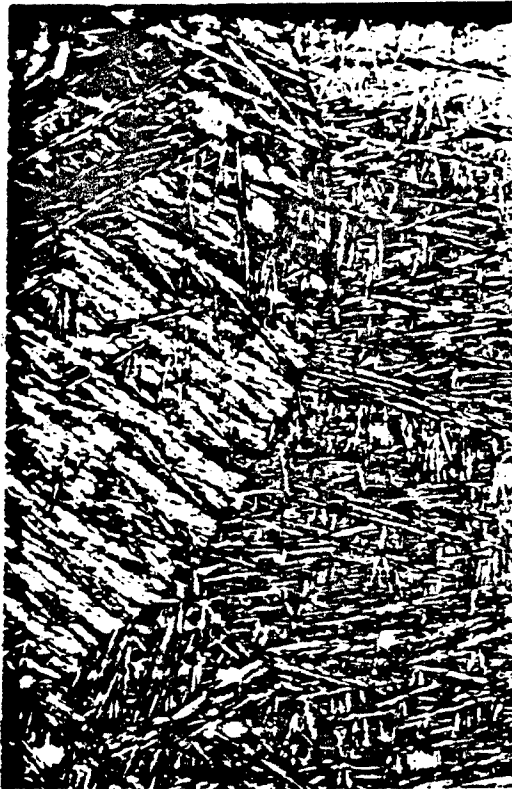


Center

50x

Figure 43. Photomicrographs of Provecut Derby Extrusions

X-7-1-10



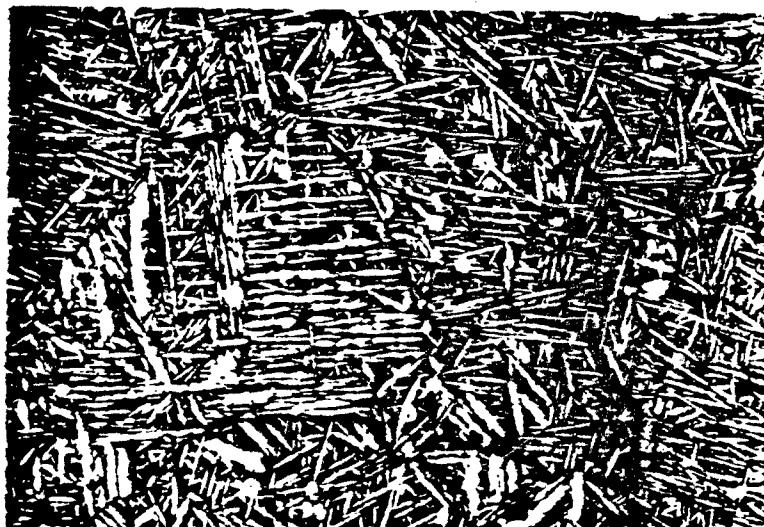
Edge

100x



Mid-radius

100x



Center

100x

X-7-1-10

Figure 44. Photomicrographs of Proveout Derby Extrusions



Edge

250x



Mid-radius

250x



Center

250x

X-7-1-10

Figure 45. Photomicrographs of Proveout Derby Extrusions.





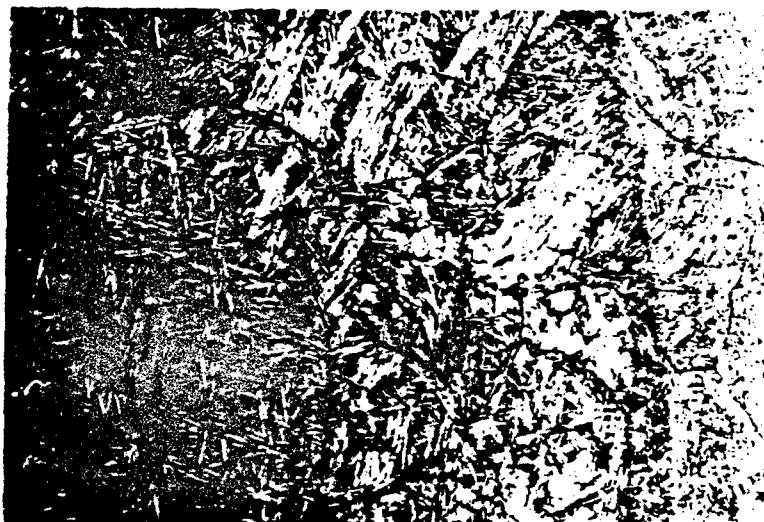
Edge

50x



Mid-radius

50x



Center

50x

X-8-5-1

Figure 46. Photomicrographs of Proveout Derby Extrusions



Edge

100x



Mid-radius

100x

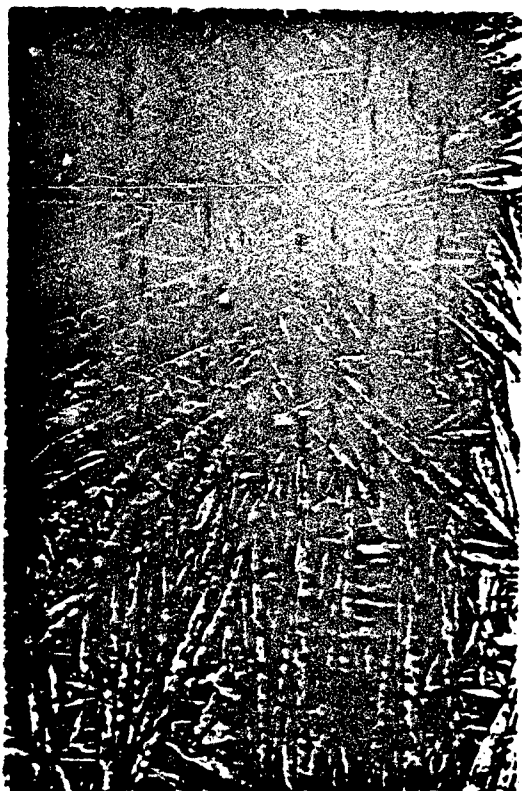


Center

100x

X-8-5-1

Figure 47. Photomicrographs of Proveout Derby Extrusions



Edge

250x



Mid-radius

250x



Center

250x

X-8-5-1

Figure 48. Photomicrographs of Proveout Derby Extrusions



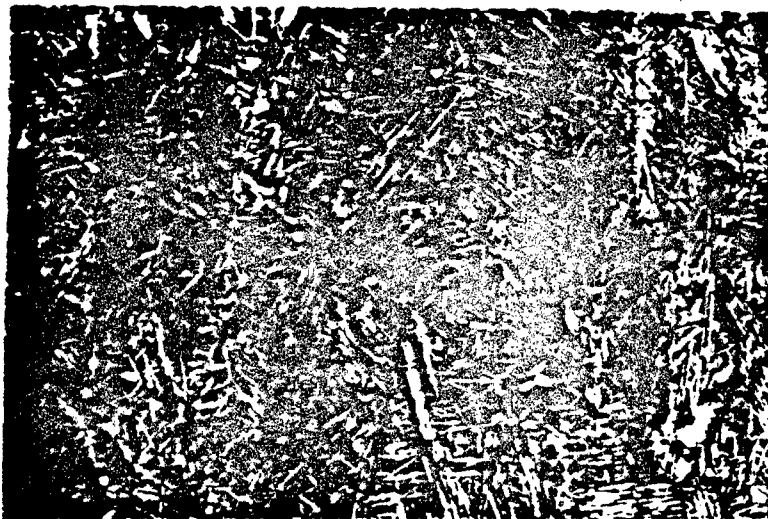
Edge

50x



Mid-radius

50x



Center

50x

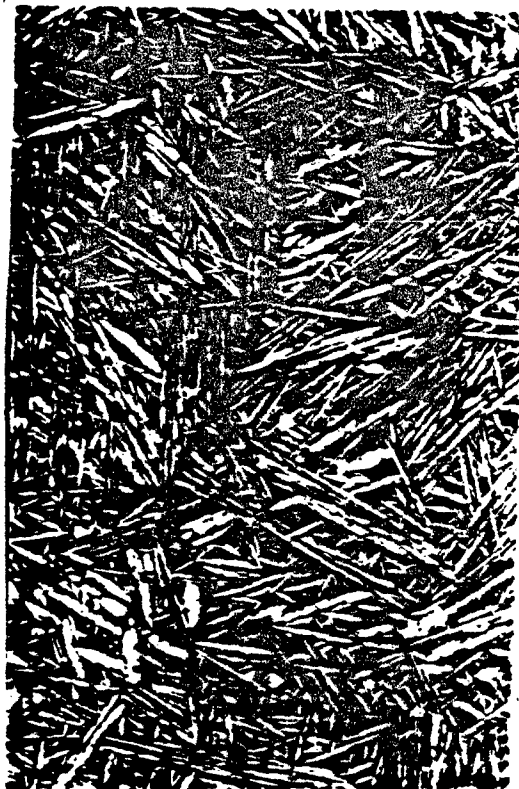
X-8-5-11

Figure 49. Photomicrographs of Proveout Derby Extrusions



Edge

100x



Mid-radius

100x



Center

100x

X-8-5-11

Figure 50. Photomicrographs of Proveout Derby Extrusions



Edge

250x



Mid-radius

250x



Center

250x

X-8-5-11

Figure 51. Photomicrographs of Proveout Derby Extrusions





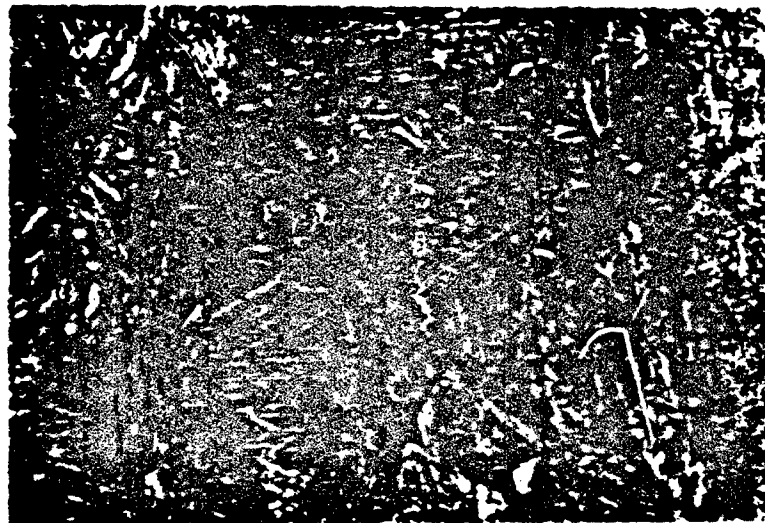
Edge

50x



Mid-radius

50x



Center

50x

X-10-1-1

Figure 52. Photomicrographs of Proveout Derby Extrusions



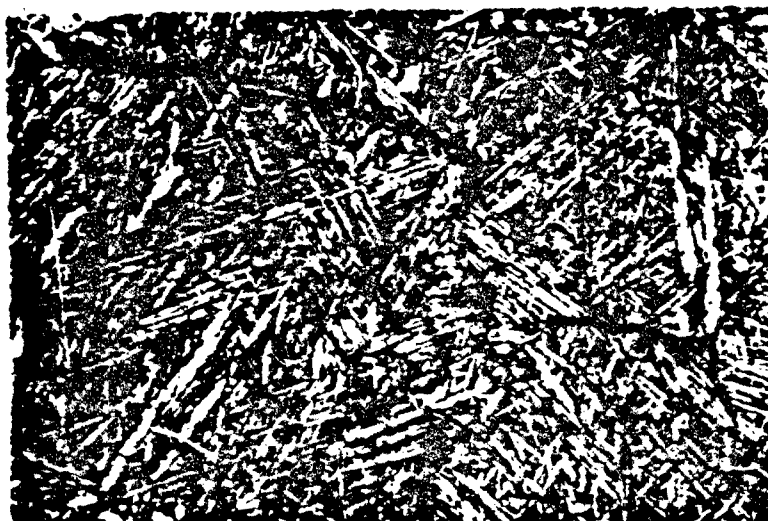
Edge

100x



Mid-radius

100x



Center

100x

X-10-1-1

Figure 53. Photomicrographs of Proveout Derby Extrusions





Edge

250x



Mid-radius

250x



Center

250x

X-10-1-1

Figure 54. Photomicrographs of Proveout Derby Extrusions



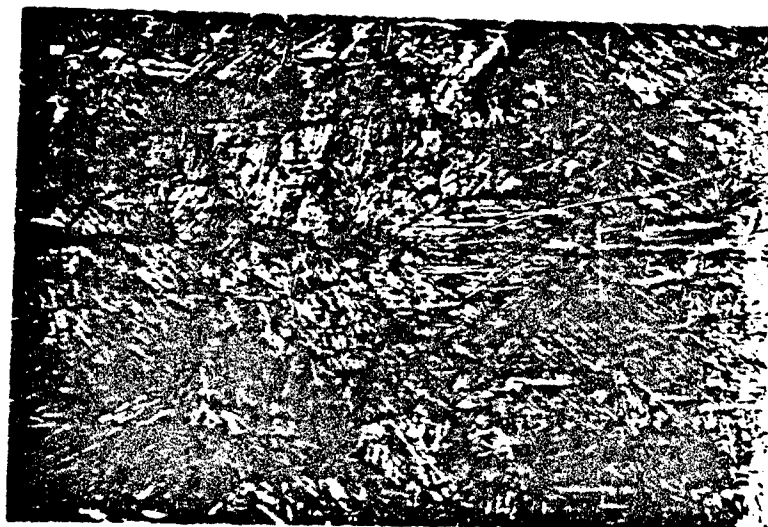
Edge

50x



Mid-radius

50x



Center

50x

X-10-1-10

Figure 55. Photomicrographs of Proveout Derby Extrusions



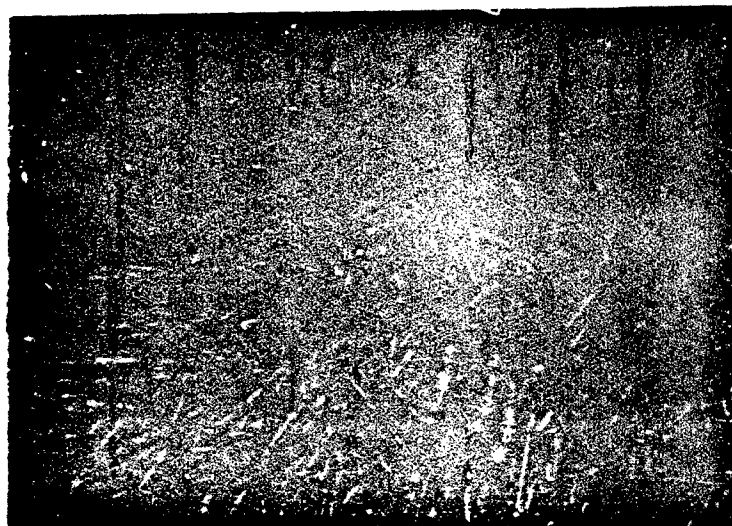
Edge

100x



Mid-radius

100x

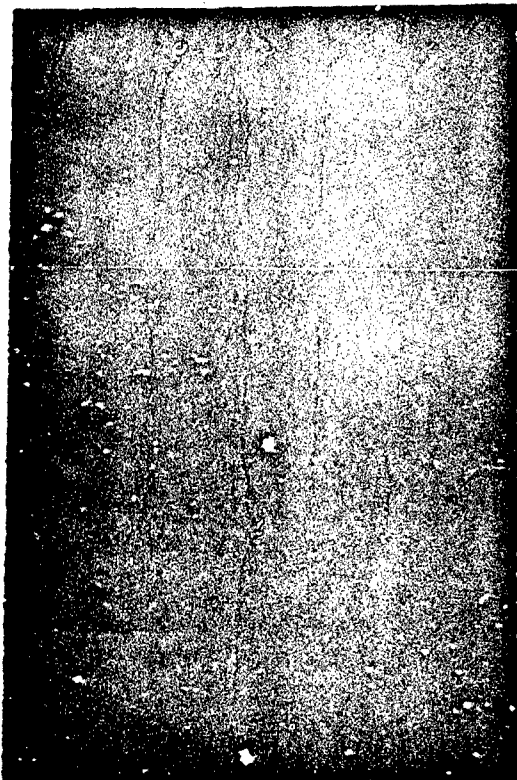


Center

100x

X-10-1-10

Figure 56. Photomicrographs of Proveout Derby Extrusions



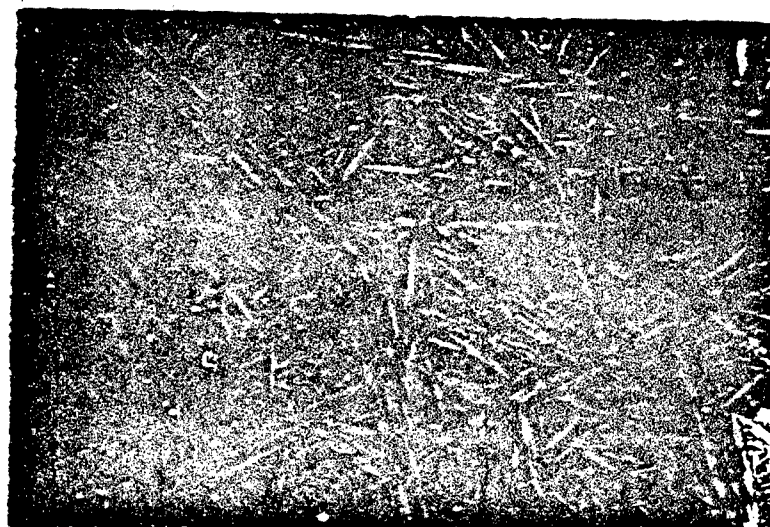
Edge

250x



Mid-radius

250x



Center

250x

X-10-1-10

Figure 57. Photomicrographs of Proveout Derby Extrusions

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